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Electroplating Wastewater Sludge Characterization

American Electroplaters' Society, Inc.
Winter Park, FL

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ELECTROPLATING WASTEWATER SLUDGE CHARACTERIZATION

by

American Electroplaters' Society, Inc.
1201 Louisiana Avenue
Winter Park, Florida 32789

and

Centec Consultants
11260 Roger Bacon Dr.
Reston, Virginia 22090

Cooperative Agreement
No. R880026-01

Project Officer

A. B. Craig, Jr.
Nonferrous Metals and Minerals Branch
Energy Pollution Control Division
Industrial Environmental Research Laboratory
Cincinnati, Ohio 45268

INDUSTRIAL ENVIRONMENTAL RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U. S. ENVIRONMENTAL PROTECTION AGENCY
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16. ABSTRACT

EPA's RCRA regulations have provisions, the Extraction Procedure (EP), for evaluating the hazardous nature of certain materials. This test method was evaluated by EPA and the American Electroplaters' Society and applied to metal finishing sludges. Concurrently, tests to chemically and physically characterize these sludges were performed.

This report provides an evaluation of the leachability of metal finishing sludges tested according to the EPA's Extraction Procedure, (EP). The results showed that many sludges were disposed segregated from acidic environments. However, when tested by the EP, many were found to leach excessively. This is partly an artifact of the test procedure but does indicate that basic sludges will redissolve in acidic environments. Questions should be directed to the Nonferrous Metals and Minerals Branch of the Industrial Environmental Research Laboratory.

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FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory-Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report provides an evaluation of the leachability of metal finishing sludges tested according to the EPA's Extraction Procedure, (EP). The results showed that many sludges were disposed segregated from acidic environments. However, when tested by the EP, many were found to leach excessively. This is partly an artifact of the test procedure but does indicate that basic sludges will redissolve in acidic environments. Questions should be directed to the Nonferrous Metals and Minerals Branch of the Industrial Environmental Research Laboratory .

David G. Stephan
Director
Industrial Environmental Research Laboratory
Cincinnati

ABSTRACT

This project was initiated to provide information concerning the physical and chemical characteristics of electroplating wastewater treatment sludges and their performance in a leaching environment. Phase I of the study characterized, physically and chemically, representative electroplating sludges, determined their response to the EPA extraction procedure (EP), and determined the sensitivity of the parameters in the EP. Phase II involved laboratory testing to determine the performance of electroplating sludges under less aggressive leaching conditions than those present in the EP. Phase III experiments were designed to validate conclusions drawn from Phase II by conducting field tests on an actual segregated metal hydroxide landfill. Twelve electroplating shops with different types of production were sampled to supply sludges for the study.

Two variables were found to have a major effect on the results obtained by the leaching procedures tested during Phase I: The composition and amount of free and interstitial wastewater present in the sludge, and the pH of the final extract. Other observations worthy of note include: After the initial flushing of interstitial and supernatent waters, the sludges do not leach heavy metal at a significant rate when leached with deionized water; due to its low pH, acidified water has an aggressive leaching effect on certain metals found in electroplating sludges; under field conditions tested, electroplating wastewater sludges do not leach significant quantities of heavy metals when disposed of in a segregated hydroxide landfill; and electroplating sludges resist leaching of heavy metals unless subjected to aggressive leaching procedures.

This report was submitted in fulfillment of cooperative agreement no. R880026-01 between the U.S. Environmental Protection Agency and the American Electroplaters' Society (AES). CENTEC Corporation performed the sampling, laboratory, engineering analyses, and report writing as a subcontractor to the AES. This report covers the period March 26, 1979 to July 29, 1980, and work was completed as of July 29, 1980.

TABLE OF CONTENTS

Section

DISCLAIMER..... ii

FOREWORD.....iii

ABSTRACT..... iv

FIGURES.....vii

TABLES.....viii

ACKNOWLEDGEMENT..... ix

1 INTRODUCTION..... 1

2 CONCLUSIONS AND RECOMMENDATIONS..... 3

2.1 Conclusions..... 3

2.2 Recommendations..... 6

3 DISCUSSION OF PHASE I EXPERIMENTS..... 8

3.1 Introduction..... 8

3.2 EPA Extraction Procedure..... 11

3.3 Experiment I - Effect of pH..... 14

3.4 Experiment II - Effect of pH..... 16

3.5 Experiment III - Volume of Extraction Water..... 29

3.5 Experiment IV - Reproducibility..... 30

3.7 Experiment V - Temperature..... 35

3.8 Experiment VI - ASTM Extraction Procedure..... 35

3.9 Experiment VII - Age of Sludge..... 39

3.10 Experiment VIII - Total Metal Content..... 40

3.11 Experiment IX - Anion Content..... 42

3.12 Experiment X - X-Ray Diffraction..... 44

3.13 Experiment XI - Centrate Analysis..... 44

3.14 Experiment XII - Filtration Versus Centrifugation..... 47

3.15 Experiment XIII - Organic Content of Sludge..... 49

3.16 Other Related Studies..... 49

4 DISCUSSION OF PHASE II EXPERIMENTS..... 52

4.1 Introduction..... 52

4.2 Effect of Head Pressure on Dyanamic Testing..... 54

4.3 Dynamic Testing with Acidified Water..... 66

5 DISCUSSION OF PHASE III EXPERIMENTS..... 77

APPENDIX A
ANALYTICAL QUALITY CONTROL..... 81

APPENDIX B
BASICITIES OF METAL IONS..... 83

APPENDIX C
Plant Wastewater Treatment Systems..... 85

FIGURES

Number		Page
1	Metals Concentration as a Function of pH - Plant Number 1A	23
2	Metals Concentration as a Function of pH - Plant Number 2A	23
3	Metals Concentration as a Function of pH - Plant Number 3A	24
4	Metals Concentration as a Function of pH - Plant Number 4A	24
5	Metals Concentration as a Function of pH - Plant Number 5A	25
6	Metals Concentration as a Function of pH - Plant Number 6A	25
7	Metals Concentration as a Function of pH - Plant Number 7A	26
8	Metals Concentration as a Function of pH - Plant Number 8A	26
9	Metals Concentration as a Function of pH - Plant Number 9A	27
10	Metals Concentration as a Function of pH - Plant Number 10A	27
11	Metals Concentration as a Function of pH - Plant Number 11A	28
12	Metals Concentration as a Function of pH - Plant Number 12A	28
13	Dynamic Testing Apparatus	53

TABLES

Number		Page
I	Description of Experiments	9
2	Plant Description	10
3	Slude Description	13
4	Effect of pH	17
5	Effect of pH	19-22
6	Volume of Extraction Water	31
7	Reproducibility of EP	32
7A	Reproducibility of EP	33
8	Independent Laboratory Confirmation	34
9	Temperature Study	36
10	The ASTM Leaching Test 1	38
11	Analysis of Dry Solids	41
12	Anion Analysis	43
13	Identification of Dry Solids	45
14	Centrate Analysis	48
15	Comparison of Centrifugation vs. Filtration	50
16	Organic Content	51
17	Leachate Characteristics from Dyamic Testing of electroplating Sludge with Deionized Water at Different Head Pressures	56-62
18	Conductivity & pH of Acidified Water Used for Leaching in Dynamic Testing	67
19	Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Acidified Water	70-75
20	Characterization of Samples Taken from Segregated Landfills	79

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For further information please contact the Nonferrous Metals and Minerals Branch (513) 684-4491.

SECTION 1

INTRODUCTION

The problem of disposal and handling of solid wastes from electroplating wastewater treatment systems has been difficult and complex. This has been at least partially due to the void of knowledge concerning the physical and chemical characteristics of the sludges and their performance in a leaching environment. The project discussed here was initiated to help fill this void of information and in the process establish a method which may be applicable to other industry groups interested in learning more about their wastes.

The American Electroplaters' Society (AES) and the U.S. Environmental Protection Agency (EPA) Industrial Environmental Research Laboratory (IERL) cooperated in this three-phase study of sludges from electroplating wastewater treatment systems. Phase I was designed to characterize, physically and chemically, representative sludges of this type, to test their response to the Extraction Procedure[1] (EP) proposed by the EPA as the primary test of the toxic nature of wastes under RCRA, and to determine the sensitivity of the parameters in the EP. Phase II involved laboratory testing to determine the performance of electroplating sludges under less aggressive leaching conditions than those

[1] Federal Register, December 18, 1978, pp. 58956-7, Vol. 43, No. 243.

present in the EP. Phase III experiments were designed to validate the conclusions drawn from Phase II by conducting field tests on actual segregated metal hydroxide landfills. The ultimate goal of the study was to provide a data base which would make possible the characterization of the dissolution of toxic metals in electroplating sludges in a segregated hydroxide landfill environment. This will hopefully lead to environmentally safe landfill designs while avoiding the economic problems associated with overdesign.

This report presents the data obtained as a result of experiments conducted during Phases I, II and III of this study, along with conclusions based on these results.

SECTION 2

CONCLUSIONS & RECOMMENDATION

2.1 CONCLUSIONS

Two variables were found to have a major effect on the results obtained by the leaching procedures tested during Phase I:

- o The composition and amount of interstitial wastewater present:

  Sludges with high solids concentration consistently performed better in the leaching tests. The EPA Extraction Procedure dictates that the initial filtrate be added to the EP Extract fluid before metal analyses are conducted. High metals levels in the filtrate, which would wash out very rapidly and not be a continuous problem, combined with low solids content in the sludge, may result in higher metal levels in the EP Extract fluid than the solids portion of the sludge alone would account for. It is therefore likely that dewatering of low solids content sludges would improve the results of the leaching test.

- o The pH of the final extract:

  Metal hydroxides follow a generally predictable solubility curve with pH; when the effects of the interstitial water are taken into consideration, the sludges tested generally followed this relationship. Thus, with a pH maintained at 5.0, as in the EPA Extraction Procedure, relatively

large amounts of metals are and would be expected to be solubilized; at values closer to neutral or those produced by the natural alkalinity of the sludge, greatly reduced amounts of metal are leached. This is significant because in a segregated hydroxide landfill, the alkalinity of the sludges would, under most circumstances, maintain the pH much higher than the 5.0 used to simulate the conditions of co-disposal with organic materials.

- o Mass transfer limitations:

  This effect, which is not measured by any equilibrium type of leaching test, tends to reduce further the amount of metals transferred to leachate in a segregated landfill.

  Dynamic testing has indicated lower metal values in the leachate than reported by the EPA-EP, an equilibrium type of test. Dynamic tests with different head pressures and water flow rates support the conclusion that the leaching rate is controlled by the diffusion rate of the metal ions through the sludge mass and that the quantity of water used has little impact on the quantities of metal leached.

Additional observations which have been drawn from all three phases include:

- o After the initial flushing of interstitial and supernatent waters, electroplating wastewater sludges do not leach heavy metals at a significant rate when leached with deionized water.

- Due to its low pH, acidified water has an aggressive leaching effect on certain metals found in electroplating sludges. However, of the hazardous metals only Cd was leached at a substantially higher rate with acidified water than with deionized water and was present in only one of the sludges tested with acidified water. Since one sample cannot be used to draw a conclusion, further testing should be conducted to verify this observation.
- Under field conditions tested, electroplating wastewater sludges do not leach significant quantities of heavy metals when disposed of in a segregated hydroxide landfill. Leachate collected from an actual segregated landfill contained metal concentrations well below EPA interim primary drinking water standards for all metals currently proposed as being hazardous under RCRA--even before dilution. This applies directly only to the conditions studied but indicates that a properly designed and managed disposal site need not incorporate extravagant leachate collection techniques to prevent any environmental harm.
- Electroplating sludges resist leaching of heavy metals unless subjected to aggressive leaching procedures. While sludges may contain high concentrations of a metal, such as Cr, even the EPA Extraction Procedure removes only a small fraction of the contained metal.

2.2 RECOMMENDATIONS

Since continued investigation into solubilization phenomena may provide relatively inexpensive but environmentally safe methods for the disposal of metal finishing sludges, specifically through the use of segregated landfills, it is recommended that:

- An equilibrium type of laboratory test which would predict the behavior of hydroxide sludges in a segregated landfill should be developed.
- More rapid evaluation procedures suitable for use by generator and disposal site operators are needed and should be developed.
- The relationship of wastewater treatment technologies and chemicals to the leaching tendencies and stability of the sludges generated should be investigated to determine which treatment variables have the biggest impact on the leaching of heavy metals.
- Test results based on acidified water as the leaching fluid indicate that the effects of acid-rain on real world segregated hydroxide landfills should be further investigated.
- The remarkable reductions in the leaching characteristics of the one sludge allowed to age 3 months prior to being extracted indicate that this phenomenon may represent a very inexpensive technology for significantly reducing the leaching of heavy metals and therefore aging should be further tested.

- It is strongly recommended that this report be included as an addendum to the Best Engineering Judgement document which is used to select methods for disposal of electroplating solid wastes.

SECTION 3

DISCUSSION OF PHASE I EXPERIMENTS

3.1 INTRODUCTION

Phase I had three goals: (1) to characterize the response of representative electroplating sludges to the Extraction Procedure (EP) developed by the EPA, (2) to characterize, physically and chemically, the sludges, and (3) to determine the sensitivity of parameters contained in the EP when varied.

Table 1 describes the experiments that were run to achieve these goals.

The AES Task Force recommended a set of 12 waste treatment sludges that adequately represent the electroplating industry, along with the plant sites where the sludges could be obtained. Table 2 lists each plant code and the major processes at each plant contributing to the sludge.

During the sampling visit, an effort was made to collect readily available information concerning plant operations and wastewater handling techniques as they pertained to the sludges. Sampling was somewhat representative despite the absence of standardized sampling techniques. Where possible the sludge samples were collected as they came out of the dewatering process, although some samples were collected from dewatering beds and may have been several months old. The intent was to collect samples in the form in which they would be shipped to a disposal site.

Several of the experiments conducted were intended to be screening tests to determine if further investigation would be warranted. Insufficient data precluded firm conclusions based on these screening experiments.

Table I

Description of Experiments

Experiment	Title	Description
I	Effect of pH	Three different sludges were extracted at pH = 4, 5, 6, 7, 8, 9, 10 and 11.
II	Effect of pH	Nine additional sludges were extracted at pH = 5, 7, 8, 9 and 10.
III	Effect of Volume of Extraction Water	One sludge was extracted according to the EPA-EP, except that 5 and 10 mls of extraction water per gram of solid were used instead of 16.
IV	Reproducibility	Three sludges were extracted in duplicate to obtain some indication of the reproducibility of results obtained.
V	Effect of Temperature	One sludge was extracted at a temperature of 40°C (104°F) which represents the upper extreme allowed by the EPA-EP.
VI	ASTM Procedure	A few extractions were conducted utilizing the ASTM extraction procedures for comparison to the EPA-EP.
VII	Effect of Age of Sludge	One sludge was allowed to age for 3 months, then extracted using the EPA-EP.
VIII	Total Metal Content	The total metal content was determined for each sludge.

Table I

Description of Experiments
(Continued)

IX	Anion Content	Anions were analyzed for because of their potential impact on the solubility of metal ions.
X	X-Ray Diffraction	X-ray diffraction was run on each of the sludge samples to identify the crystalline structures present in the sludge.
XI	Centrate Analysis	The initial centrate from the EPA-EP was analyzed to determine the presence of toxic metals and their impact on the results of the EPA-EP test.
XII	Filtration versus Centrifugation	Filtration and centrifugation as separation techniques were evaluated in this test.
XIII	Organic Content	Total Carbon and Total Organic Carbon for the sludges were measured to indicate the potential for biological action.

Table 2

Plant Descriptions

Plant Code	Primary Plating Process
1	Segregated Zn
2	Segregated Cd
3	Zinc Plating and Chromating
4	Cu-Ni-Cr on Zn
5	Al Anodizing
6	Ni-Cr on Steel
7	Multi-process Job Shop
8	Electroless Cu, Acid Cu, Ni, and Cr on Plastic
9	Multi-process with Barrel or Vibratory Finishing
10	Printed Circuits
11	Ni-Cr on Steel
12	Cd-Ni-Cu on Brass and Steel

The twelve different sludges and slurries collected by the AES/CENTEC sampling team were characterized to determine the aqueous leachability of several metallic components. The EPA Extraction Procedure (EP) was used as the primary leaching methodology. In the descriptions which detail the experiments of the Phase I study, modifications to and deviation from the EP are clearly noted. All results are presented in mg/l unless noted.

3.2 EPA EXTRACTION PROCEDURE

Because the EPA-proposed EP was used as the basic extraction procedure for the tests in this study, a general description of the EP follows to assist the reader in understanding and interpreting the data. Bear in mind that the EPA-EP was designed to simulate codisposal of industrial wastes with municipal waste in a landfill.

A 100-gram sample (minimum) of the sludge is separated into its component phases by either filtration or centrifugation.[2] The filtrate is set aside and stored at 1 to 5°C (34 to 41°F). The solid portion is prepared for extraction by reducing the particle size so that it will pass through a standard 9.5 mm (3/8") sieve. After being weighed, the solids are placed in an extraction container where 16 grams of water are added for every gram of solids.

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[2] Federal Register, May 19, 1980, pp. 33127-8, Vol. 45, No. 98. EPA eliminated the option of centrifugation as a primary separation technique shortly after completion of this study. Filtration may yield lower metal levels in some EP tests, especially if solids lighter than water are present.

This mixture is stirred sufficiently to assure that all sample surfaces are continuously brought into contact with well-mixed extraction fluid and to prevent any stratification. The pH is adjusted to 5.0 $\pm$ 0.2, using 0.5N acetic acid. The total amount of acetic acid is limited to 4 ml per gram of solid, beyond which no more may be added. The extraction is maintained between 20 to 40°C (68 to 104°F) for 24 hours. The mixture is separated as before into solid and liquid components. The liquid is diluted to a total volume equal to 20 times the weight of the initial solid material. To this is added the original filtrate from the raw sludge. This combined liquid is the Extraction Procedure Extract upon which analyses are made for toxic metal ions.[3]

The sludge samples were collected in 2-gallon Nalgene™ rectangular polyethylene carboys. Upon reaching the laboratory, the samples were stored in a refrigerator at 4°C and only withdrawn to obtain samples for the experimental study. Slurries were thoroughly mixed prior to withdrawal of samples for testing. A representative sample for each test was secured by emptying the sludge into a large tray, breaking up the solids into smaller pieces (approximately 3/8") and withdrawing portions from several sections of the sample. The unused material was returned to the carboy and stored in the refrigerator. The sludges collected for this phase of the study are described in Table 3.

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[3] Federal Register, December 18, 1978, pp. 58956-7, Vol. 43, No. 243.

Table 3

Sludge Descriptions

Plant	Initial pH	Percent Dry Solids	Apparent Density (g/ml)	Particle Density (g/ml)	Description
1	9.3	11			White liquid slurry, rubbery particles
2	9.8	6	1.08	0.76	Deep brown slurry
3	8.7	3	1.04	1.56	Yellow-brown slurry
4	8.5	7	1.04	1.06	Gray-green slurry
5	7.4	17	1.01	0.81	Gray-gritty paste, with visible water
6	10.2	34	1.12	0.66	Black paste with visible water
7	7.4	15	1.24	0.90	Green-brown paste moist, but no visible water
8	9.0	29	1.25	1.07	Blue-green, clay-like material
9	9.1	36	1.26	1.06	Gray-green hard lumps, dry appearance and quite compact
10	9.0	24	1.18	2.44	Dark muddy brown slurry
11	9.4	19			Homogeneous forest-green slurry
12	9.1	23			Dark blue-grey chunky paste

Blanks indicate tests not run

3.3 EXPERIMENT I - EFFECT OF pH

Sludges from Plants 6, 7, and 9 were the first plants sampled and were therefore selected to be characterized in this experiment. The sludges were extracted at pH 4, 5, 6, 7, 8, 9, 10 and 11. For Experiment I, 100 g (± 1.0 g) of sludge was transferred to a 2000 ml borosilicate glass beaker and 1600 g of deionized water (D.I.) added (only plastic and glass equipment are used in these tests). Any lumps of solid were broken up with a plastic stirring rod. Breaking up the lumps is a departure from the EP protocol and may have resulted in higher metal levels in the extract. It was done in an effort to improve the reproducibility of the experiment. Filtering or centrifuging was considered unnecessary for these sludges, because they had no visible free water. Finally, the solids were dispersed in the water with a Tekman Company UltraTurrax™ homogenizer. The pH of each sample was adjusted to the desired level with 0.5N acetic acid or 0.5N NaOH. The extractions were made using a Phipps-Bird™ Apparatus Model 300 equipped with borosilicate glass stirrers. All tests were run at 60 RPM. In all experiments, this agitation was adequate to keep the solids dispersed. The Phipps-Bird™ Apparatus is so constructed that up to six samples may be extracted at one time.

The pH of each solution was checked at 15-minute intervals during the first, second, third and fourth hours. The pH remained within ± 0.2 pH units of the desired value after the fourth hour when tests were run in the pH range of 5 to 10. A pH of 4.0 could not be reached or maintained in any of the extractions. In the case of pH 4, 50 ml of 8 percent acetic acid was added which

maintained a pH of 4.2 to 4.5 throughout the test. A pH of 11 was difficult to hold for more than several hours, but small additions of 0.5N NaOH maintained the level at 11.0. The sludge-water suspension was agitated for 24 hours. The beakers were then removed from the Phipps-Bird™ Apparatus, covered with a parafilm cover and allowed to stand and settle overnight. The mixtures were decanted into a 1-gallon glass bottle fitted with a Teflon™ lined cap. The residual slurry was centrifuged at 2300 RPM for 30 minutes and decanted as specified by the proposed extraction procedure. These decants were added to the first decant and diluted so that the final water weight was 20 times the original sample weight (2000 g in these three cases). The combined solution was analyzed for the following metals:

Arsenic	Barium	Copper
Lead	Cadmium	Nickel
Chromium	Mercury	Manganese
Silver	Selenium	Zinc

Metals were analyzed by atomic absorption techniques using EPA protocols. A description of the Analytical Quality Control is given in Appendix A.

Centrates from samples of Plant 6 and 9 separated rapidly, remained clear, and were easily decanted. Plant 7 sample contained a colloidal suspension which did not separate and could not be filtered through a #42 filter paper. These samples were permitted to set for several days, then decanted.

In each of the three sludges, the extraction solutions at pH 4.5 were highly colored. The other extractions remained colored to nearly the same degree as the original suspension.

Experiment I was run with a broad range of pH's to get a more complete picture of the sludge's response to pH. Experiment II was run at fewer pH levels as an economy measure and also because the extreme pH's proved to be difficult to maintain experimentally and thus were of limited value. The metals analysis for Experiment I are given in Table 4. All values are in mg/l.

3.4 EXPERIMENT II - EFFECT OF pH

The conditions and procedure for Experiment II were the same as for Experiment I. Samples from plants 1, 2, 3, 4, 5, 8 and 10, 11 and 12 were extracted at pH levels of 5, 7, 8, 9 and 10. The separation procedure of the slurry samples followed the EPA proposed methodology, with the centrifuge procedure being applied to these samples. A sample of approximately 200 g ($\pm$ 1.0 g) was weighed into a centrifuge bottle and spun at 2300 RPM for 30 minutes. The height of the solids was measured and the centrifuging repeated until solids were no longer being separated. The mixture was decanted and the decant was stored in a refrigerator.

The centrifuged solids were extracted with 16 times their weight of D.I. water for 24 hours on the Phipps-Bird™ Apparatus. After the final separation of solids and liquids, and dilution to volume, the retained initial decant, which was stored in the refrigerator, was added to the solution. The metals listed in Experiment I were analyzed for in these solutions.

The pH of each solution was checked each hour after the initial pH adjustment. In nearly all cases the pH level remained constant within $\pm$ 0.1 units of the desired pH throughout the 24-hour test.

Table 4

Effect of pH

Experiment I
(mg/l)

Hazard Level[a]		1.0	5.0	5.0				
Metal		Cd	Pb	Cr	Ni[b]	Zn[b]	Mn[b]	Cu[b]
Plant 6	pH 4	<0.01	0.027	184	170	0.6	12.5	0.1
(34% solids)	pH 5	<0.01	0.001	25.4	79.0	0.33	9.6	0.02
	pH 6	<0.01	0.002	3.64	10.0	0.03	3.8	0.04
	pH 7	<0.01	0.003	1.21	1.18	<0.01	0.22	0.02
	pH 8	<0.01	<0.001	0.05	<0.1	0.01	<0.05	<0.01
	pH 9	<0.01	<0.001	0.06	0.12	<0.01	<0.05	<0.01
	pH 10	<0.01	<0.001	0.08	<0.1	<0.01	<0.05	<0.01
	pH 11	<0.01	<0.001	0.16	<0.1	<0.01	<0.05	<0.01
Plant 7	pH 4	0.60	0.006	25.3	96.0	960	1.88	6.3
(15% solids)	pH 5	2.16	0.003	0.24	46.5	360	1.12	0.49
	pH 6	0.35	0.002	0.16	16.0	54	0.45	0.09
	pH 7	0.04	0.005	0.50	1.48	5	0.02	0.09
	pH 8	1.21	0.25	18.2	23.0	165	0.32	1.65
	pH 9	1.54	0.28	24.0	27.2	240	0.42	1.90
	pH 10	2.00	0.40	29.5	33.2	270	0.54	1.90
	pH 11	1.22	0.28	19.2	22.0	175	0.34	2.10
Plant 9	pH 4	0.04	0.020	5.38	167	148	1.0	77.5
(36% solids)	pH 5	0.03	0.010	0.32	72.5	77.5	0.55	23.1
	pH 6	<0.01	0.002	0.05	7.28	1.11	0.08	0.36
	pH 7	<0.01	0.001	0.05	0.48	0.25	<0.05	0.15
	pH 8	<0.01	0.002	0.04	0.22	0.09	<0.05	0.08
	pH 9	<0.01	<0.001	0.05	0.18	0.09	<0.05	0.08
	pH 10	<0.01	0.001	0.06	0.18	0.10	<0.05	0.08
	pH 11	<0.01	<0.001	0.09	0.26	0.15	<0.05	0.14

Note: As, Ag, Ba, Hg, and Se are at or near the detection limits for Plants 6, 7 and 9.

[a] FR 45, No. 98, pg. 33122.

[b] These metals are not controlled by RCRA.

Tests run on Plant 5 sludge at pH's of 7 to 10 contained a colloidal suspension which could not be filtered through #40 or #42 paper. The quantity of colloidal suspension increased with pH.

The results for metals considered toxic under RCRA from Experiment II are presented in Table 5. Table 5-A present results for a few metals not regulated by RCRA.

The concentration levels of metals extracted from the sludges were highly dependent upon the pH of the extraction, as is presented in Tables 4 and 5 and Figures 1 through 12. As a general rule, the metal concentrations followed what would be expected based on general chemistry, i.e, most metals were highly soluble at low pH's and decreased in solubility as pH increased, while some metals reached a minimum and began increasing in concentration again. Figures 1 through 12 clearly show the amphoteric properties some metals demonstrated during the testing.

Chromium is the metal most frequently observed in the sludges tested. While the standards regulate only total chromium, it was found as Cr^{+3}, Cr^{+6}, and possibly Cr^{+2} in different sludges. Experiment X indicates Cr^{+2} is possibly present in sludge from Plant 3. Qualitative tests for Cr^{+6} indicated its presence in sludges from Plants 2 and 4 only. Both of these sludges are low solids (6 and 7 percent, respectively) and results from Experiment XI indicate that there is enough Cr in the supernatant water of the

Table 5

Effect of pH

Experiment II

Hazard Level[a]			5.0	(mg/l) 1.0	5.0	5.0	0.2	5.0
Metal			Cr	Cd	Pb	As	Hg	Ag
1	(11% Solids)	pH 5	1.22	0.23	0.041	0.073		<0.01
		pH 7	<0.05	0.01	<0.001	0.076		
		pH 8	<0.05	<0.01	<0.001	0.035		
		pH 9	<0.05	<0.01	<0.001	0.114		
		pH 10	<0.05	<0.01	.007	0.082		
2	(6% Solids)	pH 5	1.89	126	<0.001	0.005		<0.01
		pH 7	1.68	1.42				
		pH 8	1.85	0.48				
		pH 9	1.41	0.20				
		pH 10	1.74	0.25				
3	(3% Solids)	pH 5	85.0	6.00	0.009			
		pH 7	10.4	0.13	0.045			
		pH 8	17.2	0.04	0.146			
		pH 9	20.6	0.02	0.020			
		pH 10	34.2	0.04	0.042			
4	(7% Solids)	pH 5	21.8		0.038			.02
		pH 7	10.8		0.005			.02
		pH 8	15.7		0.012			.02
		pH 9	25.4		0.011			.02
		pH 10	42.2		0.008			.02
5	(17% Solids)	pH 5	<0.01		<0.001			<0.01
		pH 7	0.18		<0.001			
		pH 8	3.67		0.031			
		pH 9	4.28		0.067			
		pH 10	1.58		0.005			
8	(29% Solids)	pH 5	400		0.032		0.0450	.02
		pH 7	1.69		<0.001		0.0395	.01
		pH 8	0.86		<0.001		0.0166	.01
		pH 9	1.34		<0.001		0.0075	.01
		pH 10	0.30		<0.001		0.0047	<.01
10	(24% Solids)	pH 5	0.12		0.88			
		pH 7	<0.05		0.075			
		pH 8	<0.05		0.055			
		pH 9	<0.05		0.082			
		pH 10	<0.05		0.058			

-Continued-

Table 5

Effect of pH
(Continued)

Experiment II
mg/l

Hazard Level[a]			5.0	1.0	5.0	5.0	0.2	5.0
Metal			Cr	Cd	Pb	As	Hg	Ag
11	(19% Solids)	pH 5	4.22	<0.01	0.004	0.035		
		pH 7	0.18		0.002	0.006		
		pH 8	0.19		0.002	0.039		
		pH 9	0.46		0.002	0.048		
		pH 10	0.35		0.002	0.052		
12	(23% Solids)	pH 5	4.85	268	0.031	<0.001		0.03
		pH 7	0.38	1.65	0.004			0.02
		pH 8	0.38	0.46	0.004			0.02
		pH 9	0.26	0.27	0.002			0.01
		pH 10	0.56	0.34	0.002			0.02

Ba, and Se were at or below detection limits.

Blanks indicate tests not run.

[a] Federal Register, May 19, 1980, pp. 33122, Vol. 45, No. 98.

Table 5 (A)

Effect of pH

Experiment II

Metals Not Defined as Toxic Under RCRA[a]

(mg/l)

Plant	Metal	Zn	Mn	Ni	Cu
1 (11% Solids)	pH 5	1110	0.15	0.62	0.04
	pH 7	81.5	<0.10	<0.10	0.03
	pH 8	6.92	<0.10	<0.10	0.02
	pH 9	1.90	<0.10	<0.10	0.05
	pH 10	6.98	<0.10	<0.10	0.01
2 (6% Solids)	pH 5	210	9.88	0.62	0.05
	pH 7	0.96	0.64	<0.10	0.02
	pH 8	1.00	0.10	<0.10	0.02
	pH 9	0.28	<0.05	<0.10	0.01
	pH 10	0.45	<0.05	<0.10	0.02
3 (3% Solids)	pH 5	912	2.54	2.08	93.8
	pH 7	9.75	1.75	<0.10	1.42
	pH 8	6.38	0.30	<0.10	1.88
	pH 9	2.75	0.25	<0.10	0.90
	pH 10	3.88	0.25	<0.10	1.40
4 (7% Solids)	pH 5	648	2.80	2.48	49.5
	pH 7	10.5	0.16	0.18	0.78
	pH 8	2.62	<0.10	0.68	0.96
	pH 9	2.38	<0.10	<0.10	0.96
	pH 10	2.00	<0.10	<0.10	0.92
5 (17% Solids)	pH 5	<0.01	0.62	9.95	<0.05
	pH 7	0.02	0.40	0.80	0.04
	pH 8	0.32	1.52	30.5	1.00
	pH 9	0.33	1.49	30.8	0.97
	pH 10	0.28	1.16	9.7	0.63
8 (29% Solids)	pH 5	0.14	1.52	375	2.99
	pH 7	0.02	0.35	1.22	0.75
	pH 8	0.02	<0.10	0.48	0.42
	pH 9	0.07	0.15	0.68	0.68
	pH 10	0.26	<0.10	0.08	0.22
10 (24% Solids)	pH 5	0.74	0.51	1.56	30.0
	pH 7	<0.01	<0.10	0.28	3.75
	pH 8	<0.01	<0.10	0.22	3.50
	pH 9	<0.01	<0.10	0.15	2.90
	pH 10	<0.01	<0.10	0.12	3.25

-Continued-

Table 5 (A)

Effect of pH
(Continued)

Experiment II

(mg/l)

Plant	Metal	Zn	Mn	Ni	Cu
11 (19% Solids)	pH 5	941	1.71	71.0	0.24
	pH 7	2.00	0.13	0.41	0.02
	pH 8	0.88	<0.10	0.20	0.02
	pH 9	0.75	<0.10	0.20	0.03
	pH 10	0.62	<0.10	0.15	0.06
12 (23% Solids)	pH 5	169	2.38	14.8	19.2
	pH 7	0.50	<0.10	<0.10	0.32
	pH 8	0.34	<0.10	<0.10	0.34
	pH 9	0.26	<0.10	<0.10	0.22
	pH 10	0.62	<0.10	<0.10	0.56

[a] Federal Register, May 19, 1980, pp. 33122, Vol. 45, No. 98.

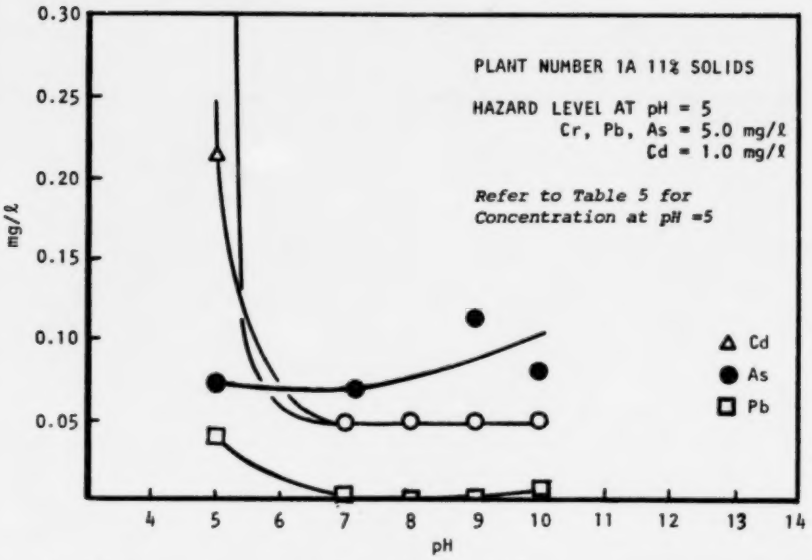

Figure 1 Metals Concentration as a Function of pH - Plant Number 1A

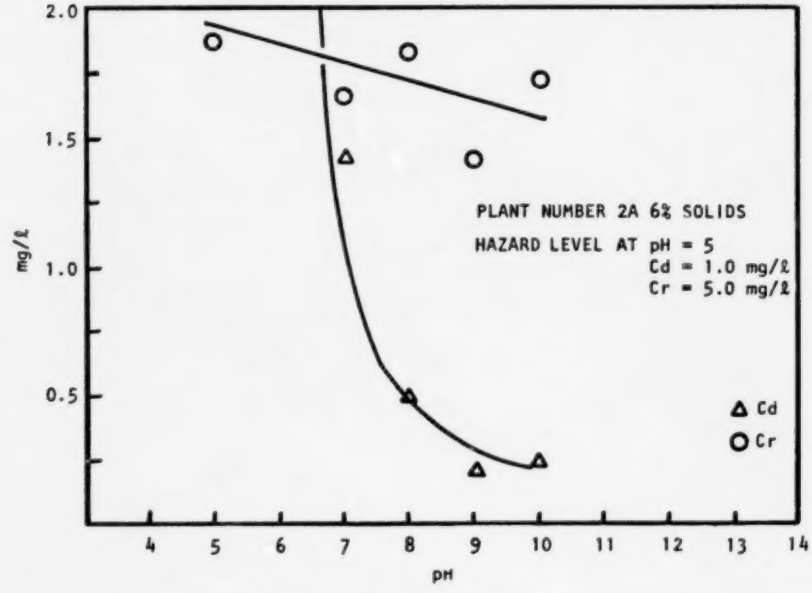

Figure 2 Metals Concentration as a Function of pH - Plant Number 2A

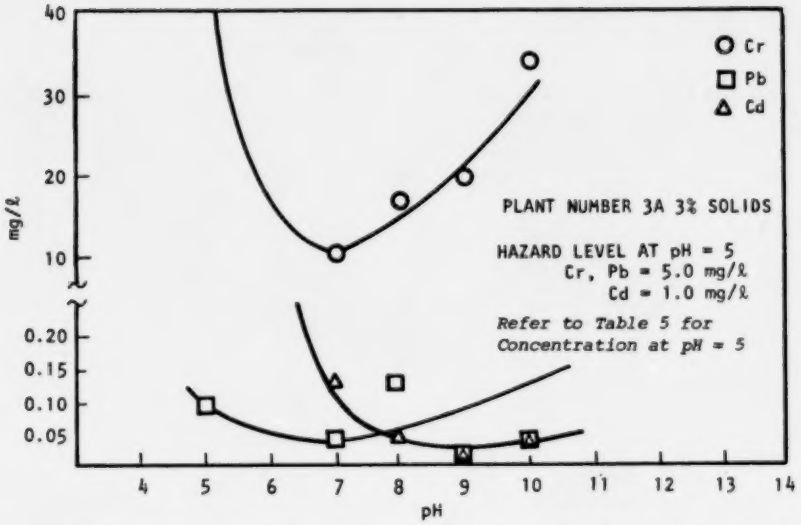

Figure 3 Metals Concentration as a Function of pH - Plant Number 3A

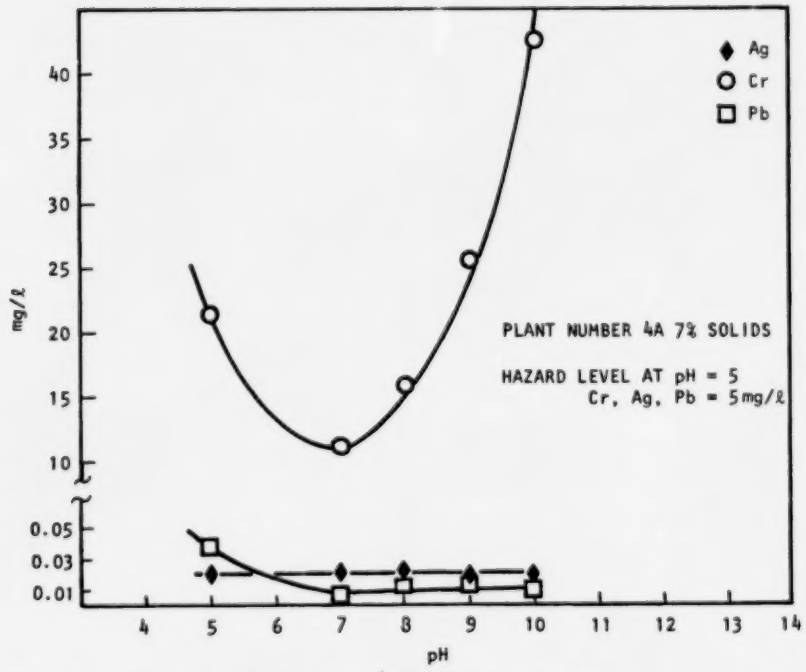

Figure 4 Metals Concentration as a Function of pH - Plant Number 4A

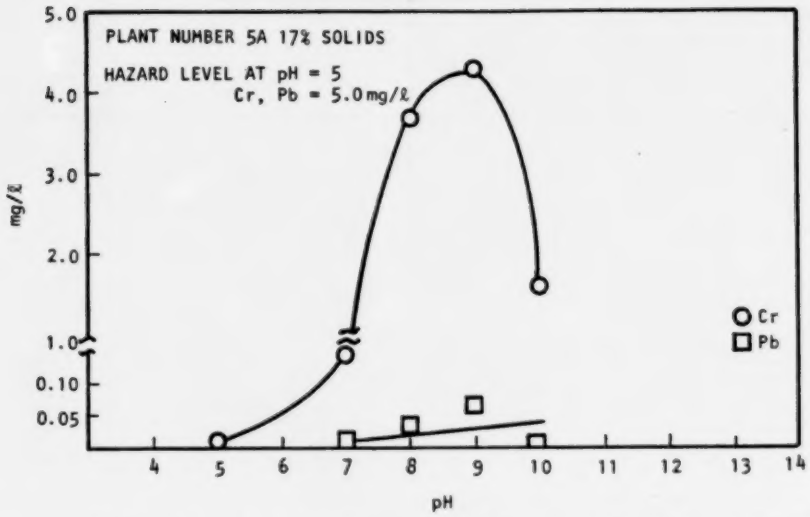

Figure 5 Metals Concentration as a Function of pH - Plant Number 5A

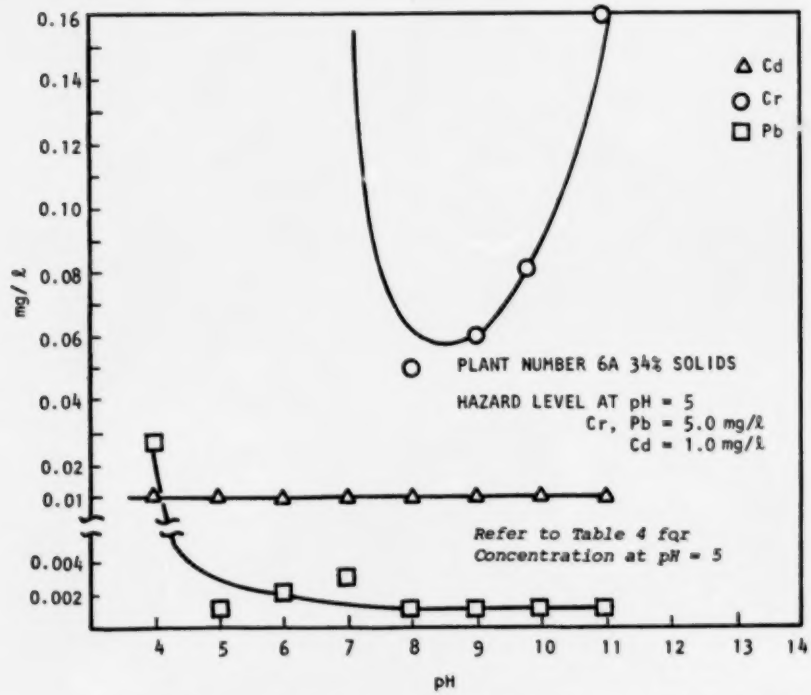

Figure 6 Metals Concentration as a Function of pH - Plant Number 6A

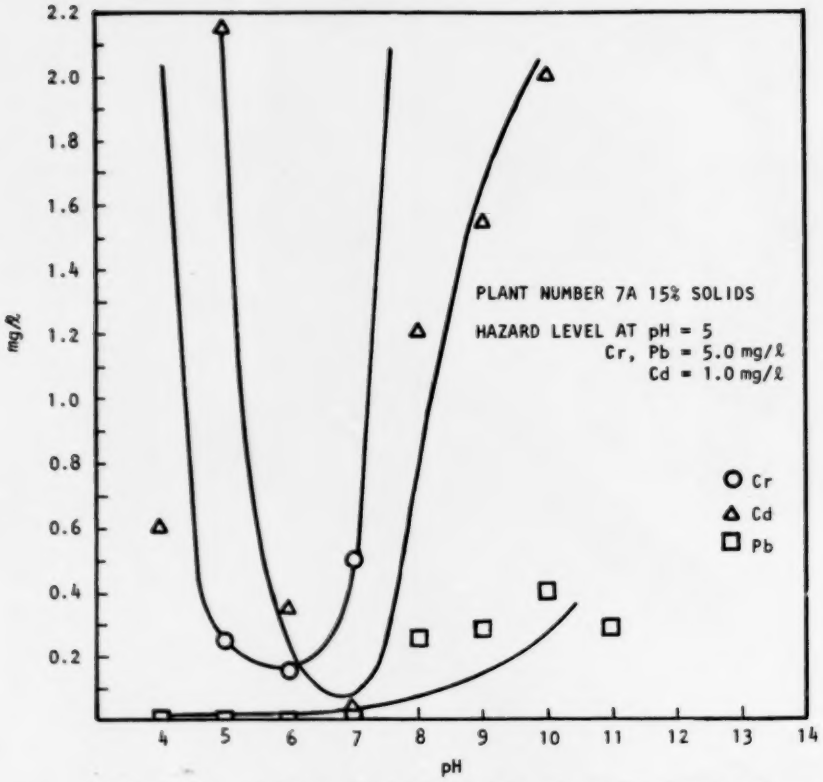

Figure 7 Metals Concentration as a Function of pH - Plant Number 7A

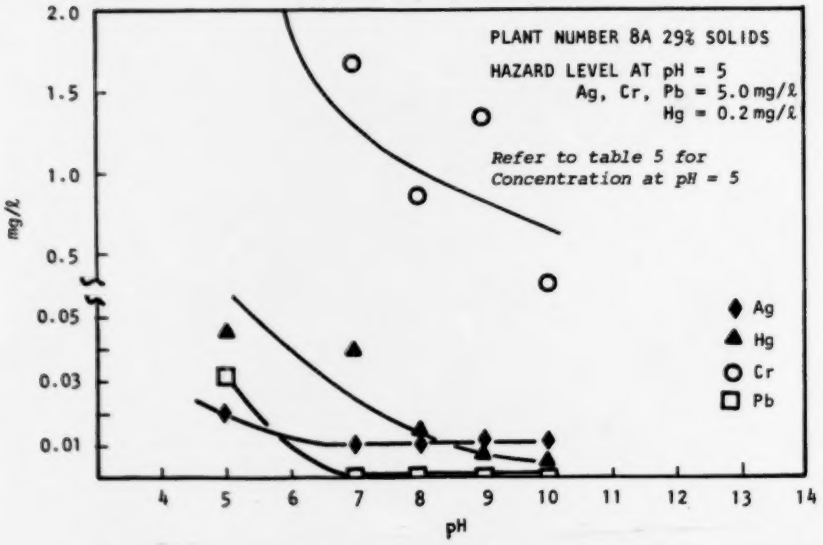

Figure 8 Metals Concentration as a Function of pH - Plant Number 8A

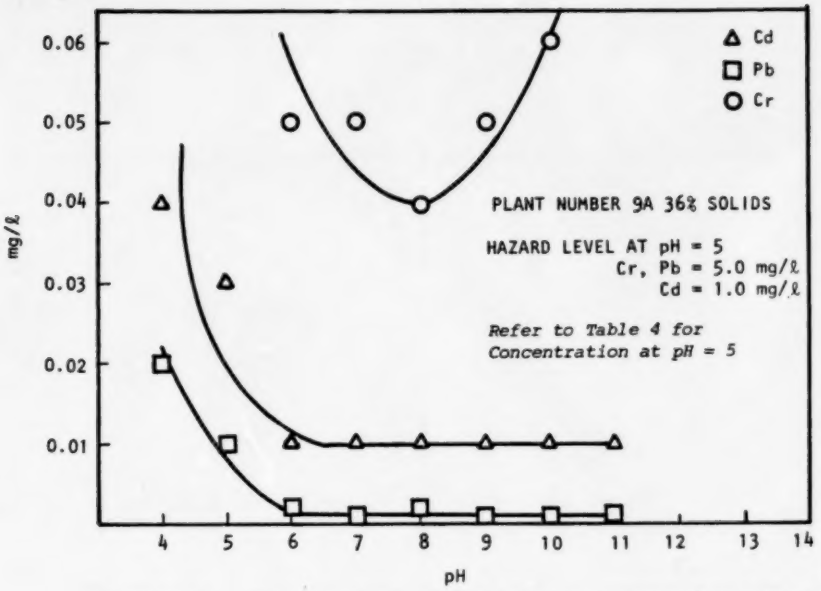

Figure 9 Metals Concentration as a Function of pH - Plant Number 9A

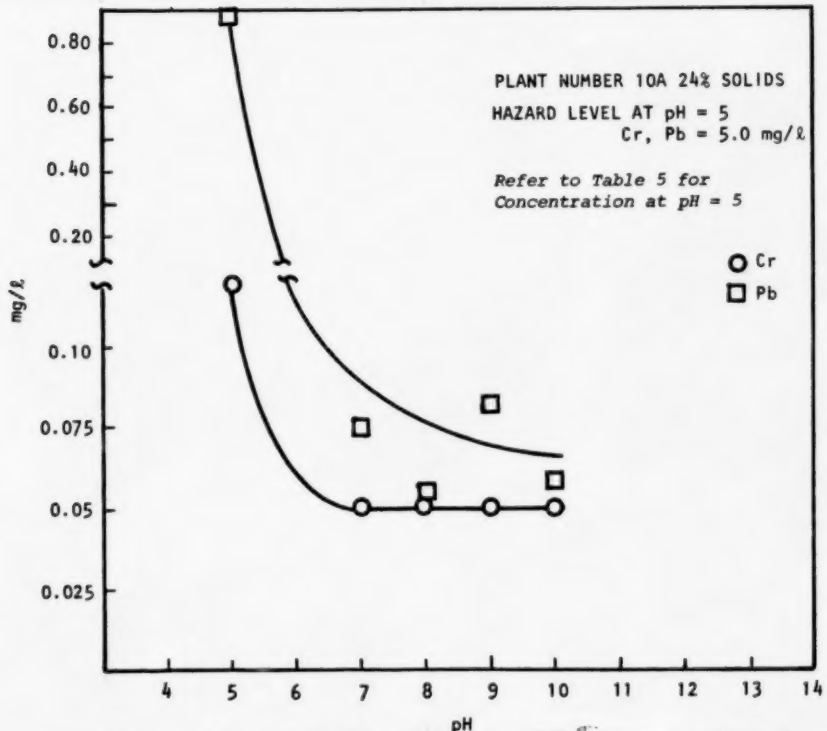

Figure 10 Metals Concentration as a Function of pH - Plant Number 10A

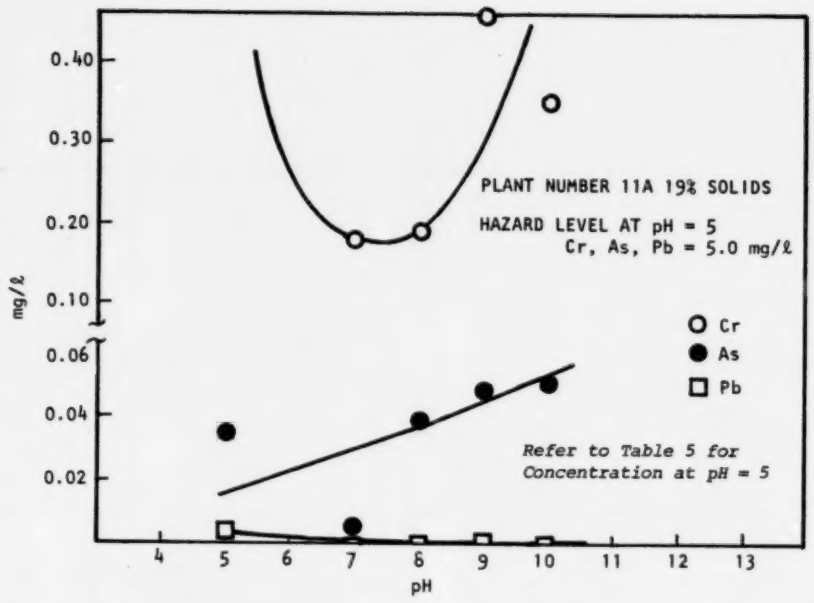

Figure 11 Metals Concentration as a Function of pH - Plant Number 11A

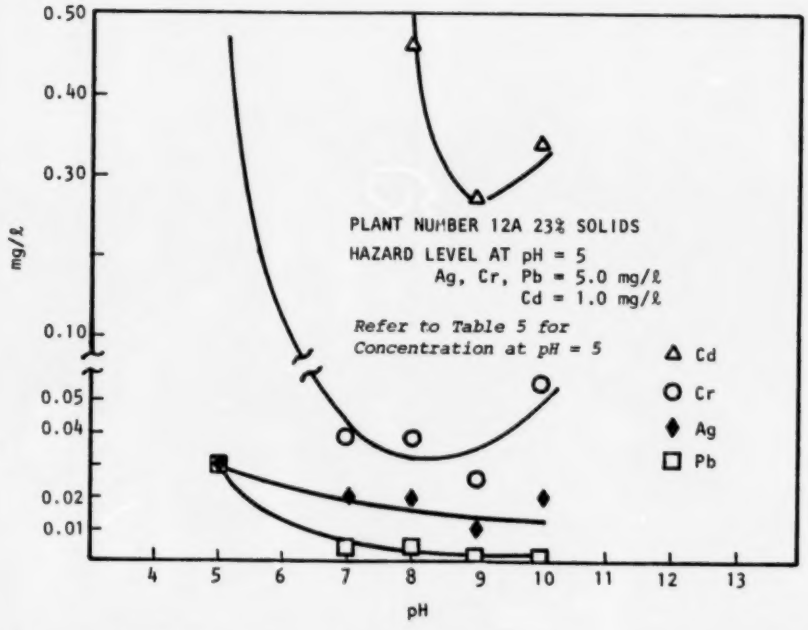

Figure 12 Metals Concentration as a Function of pH - Plant Number 12A

sludge to account for the levels of Cr found in the EP extract liquid from Plant 4. Because of the soluble nature of Cr^{+6}, it is doubtful that any significant quantities of it will be found in the solid portion of the sludge. One conclusion to be drawn from this is that some electroplating sludges will produce EP extract liquids with high metal levels due largely to the metal in the liquid and not in the solid portion of the sludge. The distinction is important because in a landfill the liquid portion flushes out with the first portion of the leachate and inclusion of this initial liquid overstates the long-term leaching from the sludge.

Centrate analysis will be discussed further under Experiment XI.

3.5 EXPERIMENT III - VOLUME OF EXTRACTION WATER

The EP states that the solids are to be extracted with a quantity of water which is 16 times the weight of the sample. In this experiment, a sludge sample from Plant 7 was extracted with quantities of water ten and five times the weight of solid, thus increasing the solids-to-water ratio. The samples were extracted at a pH of 5, 7, 8, 9, and 10. After extraction the EP extract liquid was diluted to 20 grams of liquid per gram of solids in the waste.

If the extraction were limited by solubility, the volume of extraction water should have a directly proportional effect on the metal concentration levels. That is, if the extraction ratio was

5:1, one would expect lower metal levels than at 16:1, after the extract liquid is diluted to its final 20:1. In contrast, this experiment showed that at pH 5 the volume ratio of extraction water has no or very little impact on the amount of metals extracted. On the other hand, at higher pH's the 16:1 ratio leached dramatically higher metals values than the lower ratios -- greater than can be accounted for by the final dilution to 20:1. One hypothesis is that at higher pH's the lower extraction ratios do not provide adequate liquid-solid contact to effect equilibrium during the 24-hour extraction period. At pH = 5 the same amount of metal is being extracted without regard for the extraction ratio. It would be tempting to conclude that all of the leachable metal is being extracted at pH = 5 except that we know this is not the case. A satisfactory explanation of these phenomena has not been developed. The results are given in Table 6.

3.6 EXPERIMENT IV - REPRODUCIBILITY

Sludges from Plants 7, 9 and 2 were extracted a second time at pH 5, 7, 8, 9 and 10, and the resultant solutions analyzed for those metals which were found in measurable amounts in Experiment I. There was a time lag of a few weeks between the duplicate extractions but sludge from the same batches were used. These data are given in Tables 7& 7A. Samples of the EP extraction liquid for two plants were submitted to two independent laboratories for verification of CENTEC's analytical accuracy. Both laboratories reported generally excellent concurrence, except for the Hg measurement, verifying the accuracy and reproducibility of the analytical results. Table 8 illustrates the independent

Table 6

Volume of Extraction Water

Experiment III

Plant 7 (15% Solids)
mg/l

pH	Dilution	Pb	Cr	Cd
5	5	<0.001	0.65	1.91
	10	<0.001	0.24	2.22
	16	0.003	0.24	2.16
7	5	<0.001	0.32	0.02
	10	<0.001	0.09	0.02
	16	0.005	0.50	0.04
8	5	0.003	1.84	0.11
	10	0.005	2.58	0.11
	16	0.25	18.2	1.21
9	5	0.004	1.68	0.08
	10	<0.001	1.24	0.07
	16	0.28	24.0	1.54
10	5	0.03	4.12	0.28
	10	0.008	3.99	0.22
	16	0.40	29.5	2.00

Table 7

Reproducibility of EP

Experiment IV

mg/l

	Cd		Pb		Cr	
Plant 2	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2
pH 5	126	114	<.001	.002	1.9	4.79
pH 7	1.42	2.75	<.001	<.001	1.7	1.3
pH 8	.48	.28	<.001	<.001	1.8	1.0
pH 9	.20	.18	<.001	<.001	1.4	1.2
pH 10	.25	.22	<.001	<.001	1.7	1.5
Plant 7	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2
pH 5	2.16	1.26	.003	<.001	.24	.42
pH 7	.04	.10	.005	<.001	.50	1.64
pH 8	1.21	.82	.25	.13	18.5	12.0
pH 9	1.54	-	.28	.15	24.0	-
pH 10	2.0	.90	.40	.27	29.5	21.0
Plant 9	Run 1		Run 1	Run 2	Run 1	Run 2
pH 5	.03		.010	<.001	.32	.48
pH 7	<.01		.001	<.001	.05	.08
pH 8	<.01		.002	<.001	.04	.08
pH 9	<.01		<.001	<.001	.05	.08
pH 10	<.01		.001	<.001	.06	.15

Table 7A

Reproducibility of EP

Experiment IV

Metals Not Controlled by RCRA

(mg/l)

	Zn		Mn		Ni		Cu	
Plant 2	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2
pH 5	210	200	9.88	8.25	0.62	0.70	0.05	0.06
pH 7	0.96	0.84	0.64	0.67	<0.10	<0.10	0.02	0.02
pH 8	1.00	0.22	0.10	0.07	<0.10	<0.10	0.02	0.01
pH 9	0.28	0.33	<0.05	<0.05	<0.10	<0.10	0.01	0.01
pH 10	0.45	0.60	<0.05	<0.05	<0.10	<0.10	0.02	0.02
Plant 7	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2
pH 5	360	250	1.12	1.08	46.5	39.6	0.49	0.29
pH 7	5	14.2	0.02	0.05	1.48	2.74	0.09	0.26
pH 8	165	114	0.32	0.22	23.0	14.4	1.65	1.19
pH 9	240	125	0.42	0.25	27.2	16.8	1.90	1.31
pH 10	270	200	0.54	0.38	33.2	21.6	1.90	2.25
Plant 9	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2
pH 5	77.5	118	0.55	0.78	72.5	0.70	23.1	36.0
pH 7	0.25	0.30	<0.05	<0.05	0.48	0.98	0.15	0.24
pH 8	0.09	0.38	<0.05	<0.05	0.22	0.60	0.08	0.20
pH 9	0.09	0.24	<0.05	<0.05	0.18	0.54	0.08	0.20
pH 10	0.10	0.39	<0.05	<0.05	0.18	0.80	0.08	0.30

Table 8

Experiment IV

Independent Laboratory Confirmation

of Analytical Results*

Plant		CENTEC	LAB 1		LAB 2
			Run 1	Run 2	
7	Cd	0.35	0.36	0.36	0.35
	Cr	0.16	0.18	0.18	0.23
	Pb	0.002	---	---	0.005
8	Cr	1.69	1.68	1.70	1.55
	Hg	0.0395	0.0016	0.0017	---
	Pb	<0.001	---	---	0.001

*All results in mg/l. Blanks indicate tests not run.

laboratory confirmation of analytical results. The additional tests necessary to obtain statistical measures of reproducibility were precluded by funding limitations, but the agreement shown in Tables 7,7A, and 8 does indicate sufficient reproducibility and accuracy to provide a basis for the conclusions reported herein.

3.7 EXPERIMENT V - TEMPERATURE

All of the preceding experiments were conducted at room temperature, i.e., 22°C ± 1.0°C. To determine if temperature influenced the solubility of the metal ions, an extraction of sludge from Plant 9 was made at 40°C at a pH of 5, 7, and 8. These pH levels were chosen because the data from Experiment I indicated that the metals are at their highest concentrations at the lower pH levels.

The sludge was prepared as described in Experiment I. The beakers were immersed in a water bath maintained at 40°C (± 1.0°C). Results are given in Table 9. Temperature appears to have had very limited impact on the results over the 18°C range tested for the one sludge tested.

3.8 EXPERIMENT VI - ASTM EXTRACTION PROCEDURE

Sludges from Plants 7 and 9 were extracted following the ASTM extraction procedure. Plant 7 sludge was extracted with D.I. water, and Plant 9 sludge extracted both with D.I. water and the acetic acid and sodium acetate buffer solution.

Table 9

Temperature Study

Experiment V
(mg/l)

Plant 9 (36% Solids)

	Pb	Cr
22°C - pH 5	0.013	0.32
pH 7	<0.001	0.05
pH 8	<0.001	0.04
40°C - pH 5	<0.003	0.10
pH 7	<0.001	0.19
pH 8	<0.001	0.24

Results from this experiment are presented in Table 10, along with relevant data from Experiment I.

The ASTM extraction procedure is considerably different from the EPA EP. Any reasonably sized sample of sludge can be used in the extraction. The sample is weighed without being separated and four times this weight of water is added for the extraction. The container is capped, shaken for 48 hours, allowed to separate, and then decanted. The decant is used for toxic metals analyses. A second test uses a pH 4.5 water, buffered with acetic acid and sodium acetate; otherwise, the test is the same. No pH maintenance of the extraction mixture is attempted.

The EPA-EP does not specify filtering the decant after separating the phases and filtering was therefore not performed in these tests. It is suggested that filtering would yield more meaningful results as it would remove most suspended solids from the extract liquid.

The pH's shown for the ASTM tests indicate the natural pH of the extraction. The test from Experiment I, which had the closest pH to the ASTM test, is shown for comparison. For both plants the results from Experiment I and the ASTM tests were very similar. The results of the EPA-EP are also shown for comparison. The ASTM extraction done on sludge from Plant 9 using buffered extraction water shows consistently lower results than the EPA-EP because the pH is not maintained in the ASTM test. While the EP is designed to simulate the hostile environment of a municipal landfill, the non-pH regulated procedure more closely resembles

Table 10

The ASTM Leaching Test 1

Experiment VI
(mg/l)

Hazard Level*	1.0	5.0	5.0
Metal	Cd	Pb	Cr
Plant 7			
ASTM (pH-7) (D.I.Water)	0.03	0.001	0.63
Experiment I (pH-7)	0.04	0.005	0.50
EP (pH-5.0)	2.16	0.003	0.24
Plant 9			
ASTM (D.I. Water) (pH-9.2)	-	<0.001	0.04
Experiment I (pH-9.0)	<0.01	<0.001	0.05
ASTM (Acetic Acid) (pH-4.5)	-	<0.001	0.12
EP (pH-5.0)	0.03	0.010	0.32

*For EPA-EP test results only. Federal Register, May 19, 1980, pp. 33122, Vol. 45, No. 98.

the environment of a segregated hydroxide landfill where only hydroxides or carbonates are stored and no acid environment exists.

When comparing EPA-EP and ASTM results it is important to keep in mind the differences in the dilution factor which will affect the final concentrations. The ASTM dilution factor increases as the percent dry solids decreases, while the opposite is true for the EPA-EP. For example, 100 grams of sludge with 25 percent dry solids would have 25 g of dry solids. The ASTM procedure would add 400 g of liquid for a total of 475 g of liquid, yielding 47.5 g of liquid per g of dry solids. After filtering for use in the EPA-EP, the same material would yield about 50 g of wet solids (assuming that the sludge is dewaterable to 50 percent dry solids) which would be diluted with 1000 g of liquid for a total of 1090 g of liquid, or a rate of 109 g of liquid per g of dry solids. At 5 percent dry solids, the dilution factors for the EPA-EP and the ASTM method would be 51 and 99 respectively. At 10 percent solids, the dilution factor is equal for both extraction procedures.

3.9 EXPERIMENT VII - AGE OF SLUDGE

A 1000 g sample of Plant 7 sludge was set aside in an open beaker to age for several months. Extractions at pH 5 and 7 were run on

a portion of the sample after 3 months. The results after 3 months aging were rather dramatic, as illustrated below:

Results of Aging of Plant 7's Sludge

		Cd	Pb	Cr
pH 5	Fresh	2.16	0.003	0.24
	Aged 3 months	0.30	0.002	<0.05
pH 7	Fresh	0.04	0.005	0.50
	Aged 3 months	0.01	0.001	0.15

The most evident change due to aging was in the dryness of the sample. One explanation for the drop in leachability of the toxic metals may be that some form of curing took place during the drying process which structurally tied up the metals, making them unavailable for dissolution. Further testing on other sludges is necessary before firm conclusions can be made.

3.10 EXPERIMENT VIII - TOTAL METAL CONTENT

Varying quantities of the several sludge samples were oven-dried to constant weight at 105°C to determine the percent dry solids. These dried solids were crushed and ground to a fine powder and retained in plastic Lab-Tech™ sample bottles. One gram samples were treated with aqua-regia, heated to drive off the nitrogen oxide, and diluted with water. Only two of the twelve samples were completely solubilized. The solution was filtered and the filtrate diluted to 150 ml. Aliquots of these solutions were analyzed for metals. All samples were analyzed for Ba, Cr, Cd, As, Ag, Hg, Se, Pb, Zn, Ni, Mn, and Cu. These data are reported for information and calculation purposes only. The metal analyses of the solids are given in Table 11.

Table 11

Analysis of Dry Solids

Experiment VIII

(% by wt.)

Plant	(% Total Solids)	Pb	Cr	Cd	Ba	Zn	Mn	Ni	Cu
1	11	*	.02	*	*	30.2	0.15	*	*
2	6	*	6.20	2.20	*	7.2	0.24	*	*
3	3	1.00	6.50	0.11	0.03	20.8	0.11	0.06	4.6
4	7	*	0.05	ND	0.06	0.42	*	4.8	2.4
5	17	*	0.17	ND	*	*	0.05	0.75	0.04
6	34	*	1.40			*	*	0.90	*
7	15	0.03	2.50	0.15		2.5	0.05	3.1	0.08
8	29	0.02	13.7	ND	*	*	0.01	5.8	4.7
9	36	*	0.57		*	1.85	0.03	4.1	1.3
10	24	0.17	0.35	*	*	0.04	*	0.02	0.21
11	19	0.09	7.92	*	0.03	14.3	0.03	2.06	*
12	23	0.03	4.89	0.05	0.01	5.95	0.03	0.90	3.64

* Detected but <0.01%

ND - Not Detected

Ag, As, Hg detected but <0.01%

Se was detected at Plants 11 and 12 but <0.01%

Blanks indicate test not run.

Because values for chromium were found in every sludge, a regression analysis was run on chromium to determine if a correlation existed between the amount of Cr in the dried sludge and the amount measured in the EP extract liquid (pH = 5). The correlation was R = 0.77, which indicates a strong relationship. R^2=.59, so variation in Cr content of sludge explains about 60 percent of the variation of Cr found in the extract. This may or may not hold for other metals (more data is needed) but the implication is that the more metal there is in the sludge, the higher will be the levels found in the EP extraction liquid.

3.11 EXPERIMENT IX - ANION CONTENT

Separate samples of the dry solids were taken for anion analyses to determine the presence of chlorides, sulfates, nitrates, nitrites, and total phosphorous. Chlorides were determined by the Volhard Titrametric method using silver nitrate; sulfate by the Barium Sulfate gravimetric method; and the nitrogen and phosphates by the Auto-Analyzer procedure. Nitrogen was determined as a diazo compound and phosphate as the phosphomolybdenum complex. Anions present in each of the sludges are shown in Table 12 as a weight percent of the dry solids.

Several attempts were made to determine the alkalinity of the electroplating sludges using the potentiometric technique on the aqueous fraction of the sample. As expected, due to the buffering capacity of the sludge, no meaningful value for alkalinity could be obtained.

Table 12

Anion Analysis

Experiment IX

(Weight percent of dry solids)

Plant	Cl^-	$SO_4^=$	NO_2+NO_3 (as N)	P
2	3.35	7	0.09	0.006
3	1.1	1.27		
4	1.85	2.25		
5	0.169	0.007		
6	<0.1	-	0.001	<0.001
7	<0.1	0.15	0.004	0.002
8	0.08	5		
9	<0.1	3	<0.001	0.005
10	0.6	7		

Blanks indicate tests not run.

3.12 EXPERIMENT X - X-RAY DIFFRACTION

X-ray diffraction was utilized in the identification of the chemical compositions of the crystalline species in the dry solids. They were determined by the Chemistry Department of North Carolina State University, Raleigh, North Carolina.

These data are given in Table 13. Amorphous materials were present in all of the sludges. The electron microprobe indicated the presence of metals in the order of the amount present. This test was run to verify qualitatively the results of the dry metals analyses. No unexpected results were obtained from this experiment.

The apparent and particle densities of the electroplating sludges were also determined. The apparent densities were determined on the "as received" sample by weighing a 500 ml sample. For the slurries, a well-mixed sample was poured into a 500 ml graduated cylinder; for the solids, the material was pushed into a graduated cylinder, which had been cut off at the 50 ml mark, and then weighed.

The particle densities were determined on a dried and crushed powder using a pycnometer with methylene chloride as the reference solvent.

Data were given earlier in Table 3.

3.13 EXPERIMENT XI - CENTRATE ANALYSIS

Considerable interest has been expressed as to how much of the soluble toxic metals are contained in the supernatant liquid of

Table 13

Identification of Dry Solids

Experiment IX

Sample#	X-Ray Diffraction	Electron Microprobe* (Elements lighter than Mg not read)
2	NaCl	Cl, Cr, Fe, K, Zn, P, S
3	CrO	Cr, Fe, Zn, Sn, Ca, Cu, Cl, S
4	$CaCO_3$, some SiO_2	Ca, Cr, Ni, Cu, Si, Fe, S, Cl
5	Amorphous	P, Ca, Al, Fe, Ni, Cr, Cu, Si
6	SiO_2, $CaCO_3$, Al_2O_3	Ca, Si, Fe, Cr, Al, Ni, S
7	Amorphous	Zn, P, Cr, Fe, Ca, Ni
8	$CaSO_4$, some $CaCO_3$	Ca, Cr, Ni, S, Cu, P, Si
9	$CaCO_3$	Ca, Si, Fe, Ni, Al, Cl, K, Cr
10	$CaCO_3$(syn), $CaSO_4$ $0.5H_2O$	Ca, Cu, S, Sn, Cr, Fe, Si

* In order of the amount present

the sludge. The following experiments were run to determine these metal levels and also to determine the impact of interstitial water quality on EP results.

Approximately 100 g of slurry was centrifuged. The supernatant layer was separated and analyzed for metals present in measurable concentrations.

Similarly, for solid samples, a weighed amount of solid sludge was mixed with an equal weight of water, thoroughly agitated, and then centrifuged. The liquid layer was separated and treated as the slurry described above. This procedure was designed to flush out the interstitial water, not to leach metals from the solid sludge. The data are given in Table 14.

The impact of interstitial and supernatant waters is illustrated by the following example where Plant 2's sludge had 4.08 mg/l Cr in its centrate, and only 6 percent solids in its sludge. If 75 grams of water were removed before the extraction, leaving 25 grams of sludge, there would be 500 g of extraction liquid. The 75 grams of decanted supernatant liquid contain 0.306 mg of Cr. When diluted by 575 grams of liquid (when the filtrate and extract are combined), the EP extract liquid would have a Cr concentration of 0.67 mg/l without anything being extracted from the solid. This would occur any time there is a low solids sludge and a supernatant water with metal concentrations of a few mg/l. The hazard limits promulgated on May 19, 1980 are ten times the proposed limits used as the reference points during the testing and, therefore, greatly reduced the concern discussed above.

Analyses were also run on the effluents from plants 11 and 12 and are presented in Table 14. It was expected that the metal levels in the effluent would be essentially the same as in the interstitial water; however, the data shows some substantial differences.

While concentrations and percents are very important in this kind of study, it may be illustrative to present some of the data in absolute amounts. The numbers below represent the actual amounts of chromium present in the sludge solids, the initial filtrate, and the extract liquid from an extraction run at pH 7. The values are based on a 100 gram sample.

Plant 2:	Component
	Cr
Dry Solids	770.00 mg
Centrate	1.00 mg
Extract	3.20 mg
Extract As Percent of Total Cr	0.5 percent

These numbers indicate that, even with an aggressive EP type of extraction at pH 7, very little material is being solubilized.

3.14 EXPERIMENT XII - FILTRATION VERSUS CENTRIFUGATION

At the time that the data for this study was gathered two methods for separating the solids and filtrate prior to and after the extraction procedure were acceptable; namely, filtration through a 0.45 micron filter or centrifuging followed by decantation. The EP extract liquids from Plant 10 at pH values of 5 and 7, which were centrifuged and decanted, were filtered through a 0.45 micron filter. The analysis of the decanted and filtered material

Table 14

Centrate Analysis

Experiment XI
(mg/l)

Hazard Level*	5.0	5.0	1.0
Plant	Cr	Pb	Cd
1	0.05		0.02
2	4.08	<0.001	0.33
3	47.5	0.014	0.01
4	1.52	<0.001	
5	<0.05		
6	0.47		
7	0.09		0.22
8	0.30		
9	0.04		
10	0.12	0.50	
11	2.19		
12	0.21		<0.01
			0.19

Plant Effluent

Plant	Cr	Cd	Pb	Ag	As
11	1.76	<0.01	0.002	0.01	0.017
12	1.22	2.02	0.001	0.09	0.003

Blanks indicate tests not run.

*Federal Register, May 19, 1980, pp. 33122, Vol. 45, No. 98.

and the non-filtered material are presented in Table 15. The differences in results are greater than analytical error can account for, and in this case filtering makes the difference between failing and passing the proposed EP. (Both would pass the EP limits actually promulgated.) More data are needed in this area to form a firm conclusion, but the indication is that filtering result in lower EP results for some sludges.

3.15 EXPERIMENT XIII - ORGANIC CONTENT OF SLUDGE

The TOC and TC of the sludges were measured to determine the organic levels present. Carbon is not unexpected in electroplating sludges because activated carbon is commonly used to purify plating baths and paper is frequently used as a sludge dewatering media. This experiment was conducted to verify that organic carbon is present only in very low levels. The data from this experiment are presented in Table 16, and do confirm the low levels expected to be present.

3.16 OTHER RELATED STUDIES

As each set of electroplating sludge samples arrived at the laboratory, a representative portion was transferred to a plastic container and sent to the Oak Ridge National Laboratory (ORNL).

ORNL extracted the sludges utilizing the EPA-EP, and analyzed for metal levels. For ORNL test results please contact the EPA Project Officer at the address shown in the acknowledgments.

Table 15

Comparison of Centrifugation vs. Filtration

Experiment XII

Plant 10 (24% Solids)

(mg/l)

Sample		Pb	Cr
pH 5	Centrifuge	0.88	0.12
	Filter	0.38	<0.10
pH 7	Centrifuge	0.08	<0.05
	Filter	0.01	<0.05

Table 16

Organic Content

Experiment XIII

Plant Code	% Total Organic Carbon	% Total Carbon
2	.04	.08
3	.17	.34
4	.32	.57
5	.26	.31
6	3.06	3.96
7	1.24	1.12
8	.14	.27
9	3.22	2.16
10	.53	.98

SECTION 4

DISCUSSION OF PHASE II EXPERIMENTS

4.1 INTRODUCTION

Phase I centered on the EPA proposed Extraction Procedure (EP) and tested its sensitivity to certain variables while at the same time characterizing electroplating sludge responses to these tests. The EPA-EP is an equilibrium type of test in which the sludge and extraction water are well mixed in a container for an extended period of time to reach a point where no further dissolution of the metals into the water will occur. The test is designed to simulate disposal of waste materials in an aggressive leaching environment. Testing done under Phase II of this study was designed to test the leaching tendencies of the electroplating sludges in an environment which is not so aggressive. The equipment designed for this testing is shown in Figure 13. By constantly supplying a fresh source of water, this test becomes a dynamic one as opposed to an equilibrium type test represented by the EPA-EP. Simplicity was desired to minimize the test variables; therefore, landfill lining materials (e.g., clay) were not used because of their complicated attenuation properties. Another important consideration was to develop a test which would generate results in a much shorter time than is usually associated with column tests.

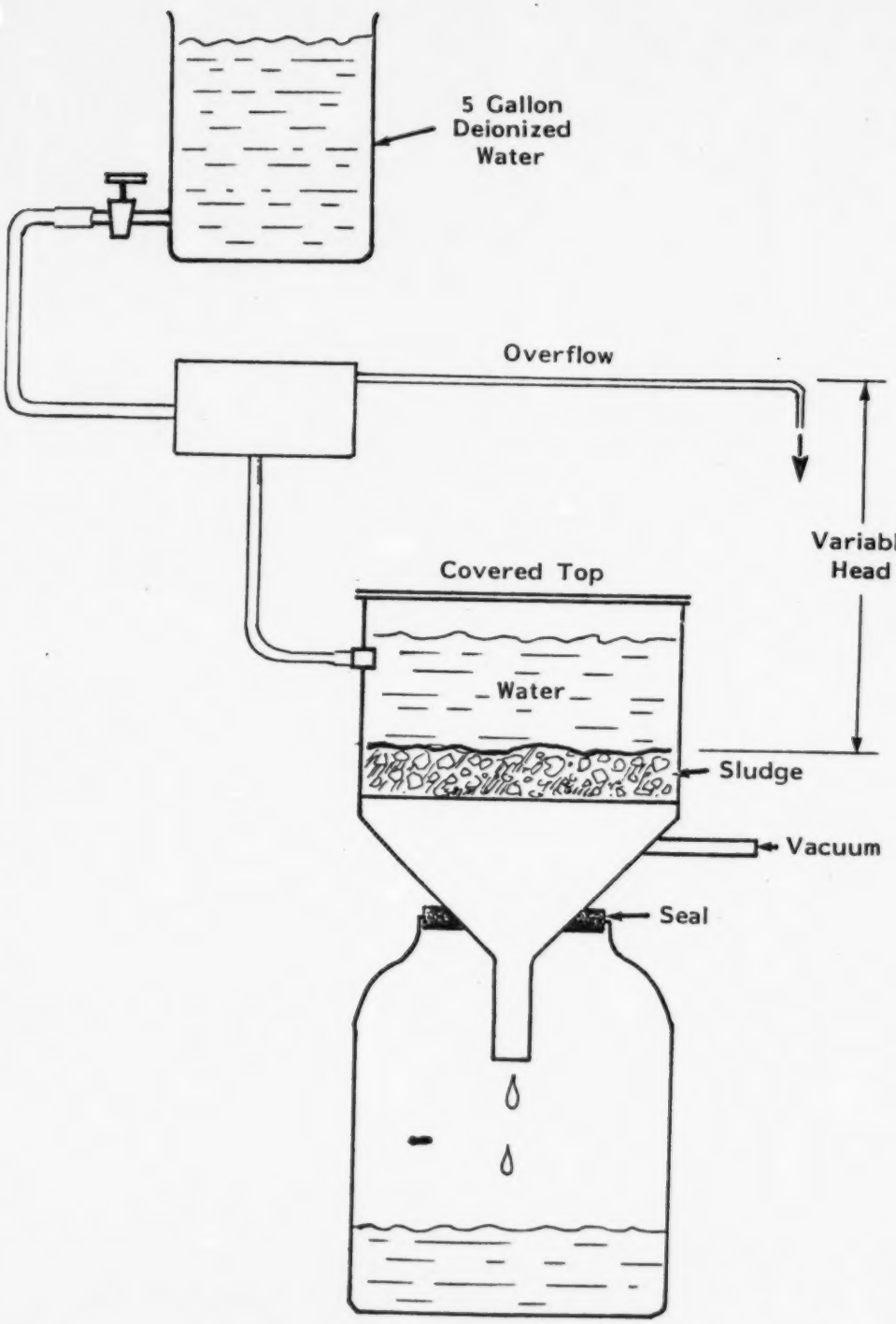

Figure 13. Dynamic Testing Apparatus

Six of the 12 sludges collected were selected for dynamic testing based primarily on the amount of sample available. One sludge was run in duplicate to evaluate reproducibility. Sludge samples were poured[4] into the apparatus and vacuum was applied to seat the sludge. The waters collected during the seating process were added back directly to the top of the sludge cake prior to starting the dynamic tests.

The sludges were subjected to a constant head of deionized water to simulate percolation of rainwater through the landfill at a constant rate. Leaching rates, while relatively constant for each sludge, varied from sludge to sludge, as would be expected due to different permeabilities. The samples were not allowed to run dry, in order to minimize the formation of surface cracks in the sludge bed which would encourage "short-circuiting" of leaching water. The leachate was measured periodically for pH and conductance to monitor the extent of leaching present. Leachate was also analyzed for metal concentrations on days 1, 2, 3, 5, 7, and every seventh day thereafter. The leachates were analyzed for all metals which were known to be in the sludge in significant quantities based on Phase I testing.

4.2 EFFECT OF HEAD PRESSURE ON DYNAMIC TESTING

The first set of dynamic tests run had a head pressure of 4 inches and the second set had a head of 11.5 inches. The

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[4]Some sludges, because of their high solids content, had to be slurried by adding water prior to seating.

most obvious result of this head difference was a change in the flow rates of DI water through the test apparatus for the respective samples. Differences in flow rates ranged from no change for the two different heads to an increase by a factor of 10 on specific days. These differences cannot, however, be entirely attributed to the different head pressures, because the effectiveness of the sludge seating varied from sample to sample. Flow rates tended to stabilize after the first day and for the low head tests ranged from approximately 66 to 2000 g/day[5] (equivalent to 0.3 to 9.7 inches of rain/day) and for the high head from 300 to 5000 g/day (equivalent to 1.5 to 24.3 inches of rain/day). The flows of leaching water for each test are shown in Table 17 and are presented as the amount of leachate collected. The wide range of flow rates from sludge to sludge is primarily a function of the permeability of the sludge and the thickness of the cake. Efforts were made to keep the cake thickness for each sludge approximately the same (0.75 inches) to minimize the impact of the thickness variable.

Table 17 also presents the pH and conductivity measurements which were taken during the testing. As would be expected with deionized water (DIW) passing through a highly buffered alkaline sludge, the initial pH readings of the leachate were initially quite high (typically in the range of 8.0 to 9.5) dropping off slowly over the duration of the tests. Even

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[5]Flow rates were measured by collecting the leachate in bottles and weighing it periodically.

Table 17

Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Deionized Water at Different Head Pressures

Plant 4

Day	pH A	pH B	Conductivity (μmhos/cm) A	Conductivity (μmhos/cm) B	Amount Collected (g/day) A	Amount Collected (g/day) B
0-1	10.35	9.32	3800	580	913	4450
1-2	10.3	9.20	300	820	1920	4953
2-3	9.9	9.10	160	58	2627	3211
3-5	9.5	9.05	100	55	1597	5280*
5-7	9.8	9.50	90	47	2234	5621
7-14	9.8	8.95	70	33	2207	5312*
14-21	9.4		50		2005	

Day	As, EP = <0.001 mg/l: mg/l A	mg/l B	μg/day A	μg/day B	Ba, EP = <1.0 mg/l: mg/l A	mg/l B	μg/day A	μg/day B
0-1	0.008	<0.001	7.3	NM	<1.0	<1.0	NM	NM
1-2		<0.001		NM		<1.0		NM
2-3		<0.001		NM		1.3		4174
3-5		<0.001		NM		<1.0		
5-7	<0.001		NM		<1.0		NM	
7-14	<0.001		NM		<1.0		NM	
14-21	<0.001		NM		<1.0		NM	

Day	Cr, EP = 21.8 mg/l: mg/l A	mg/l B	μg/day A	μg/day B	Pb, EP = 0.038 mg/l: mg/l A	mg/l B	μg/day A	μg/day B	Cu, EP = 49.5 mg/l: mg/l A	mg/l B	μg/day A	μg/day B
0-1	1.01	0.15	922	668	<0.001	<0.001	NM	NM	0.06	<0.01	55	NM
1-2	0.14	0.08	269	396		0.001		4.9	0.02	<0.01	38	NM
2-3	0.12	0.08	315	257		0.001		3.2	<0.01	<0.01	NM	NM
3-5	0.12	0.08	192	>422		<0.001		NM	<0.01	<0.01	NM	NM
5-7	0.08	0.08	179	450	<0.001		NM		<0.01		NM	
7-14	0.08	0.12	177	>637	<0.001		NM		<0.01		NM	
14-21	0.08		160		<0.001		NM		<0.01		NM	

Day	Mn, EP = 2.8 mg/l: mg/l A	mg/l B	μg/day A	μg/day B	Ni, EP = 2.48 mg/l: mg/l A	mg/l B	μg/day A	μg/day B	Zn, EP = 648 mg/l: mg/l A	mg/l B	μg/day A	μg/day B
0-1	<0.10	<0.10	NM	NM	1.40	0.22	1278	579	<0.01	<0.01	NM	NM
1-2		<0.10		NM	0.15	<0.1	288	NM	<0.01	<0.01	NM	NM
2-3		<0.10		NM	<0.10	<0.1	NM	NM	<0.01	<0.01	NM	NM
3-5		<0.10		NM		<0.1		NM	<0.01	<0.01	NM	NM
5-7	<0.10		NM		<0.10		NM		0.03		67	
7-14	<0.10		NM						<0.01		NM	
14-21	<0.10		NM						<0.01		NM	

A = Head pressure = 4 inches
B = Head pressure = 11.5 inches
NM = Not Meaningful and therefore not calculated
Blanks indicate test not run
*This is a minimum value because the leachate collector overflowed
Note: < means below detectable limits

Table 17

Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Deionized Water at Different Head Pressures

Plant 5

Day	pH A	pH B	Conductivity (μmhos/cm) A	Conductivity (μmhos/cm) B	Amount Collected (g/day) A	Amount Collected (g/day) B	As, EP = <0.001 mg/l: mg/l A	As: mg/l B	As: μg/day A	As: μg/day B	Cr, EP = <1.01 mg/l: mg/l A	Cr: mg/l B	Cr: μg/day A	Cr: μg/day B
0-1	8.3	8.98	440	1120	67	696	0.048	0.006	3.2	4.2	<0.05	<0.05	NM	NM
1-2	8.4	8.10	290	610	271	370		0.001		.4	<0.05	0.05	NM	18
2-3	8.4	8.10	175	352	65	317		<0.001		NM	0.06	<0.05	3.9	NM
3-5	8.2	7.65	93	281	66	293		<0.001		NM	0.06	<0.05	4.0	NM
5-7	8.1	7.65	95	252	67	280	0.002		0.1		0.07		4.7	
7-14	8.2	7.60	60	162	66	268	0.001		0.07		0.05		3.3	
14-21	7.9		390		64		0.003		0.2		<0.05		NM	

Day	Pb, EP = <0.001 mg/l: mg/l A	Pb: mg/l B	Pb: μg/day A	Pb: μg/day B	Cu, EP = <0.05 mg/l: mg/l A	Cu: mg/l B	Cu: μg/day A	Cu: μg/day B	Mn, EP = 0.62 mg/l: mg/l A	Mn: mg/l B	Mn: μg/day A	Mn: μg/day B
0-1	<0.001	<0.001	NM	NM	0.01	0.02	0.03	14	<0.10	<0.10	NM	NM
1-2		0.001		0.4		0.09		33	<0.10	<0.10	NM	NM
2-3		<0.001		NM		0.07		22	<0.10	<0.10	NM	NM
3-5		<0.001		NM		0.03		8.8		<0.10		NM
5-7	<0.001		NM		0.08	0.02	5.4	5.6	<0.10		NM	
7-14	<0.001		NM		0.07	0.01	4.6	2.7	<0.10		NM	
14-21	<0.001		NM		0.06		3.8		<0.10		NM	

Day	Ni, EP = 9.95 mg/l: mg/l A	Ni: mg/l B	Ni: μg/day A	Ni: μg/day B	Zn, EP = <0.01 mg/l: mg/l A	Zn: mg/l B	Zn: μg/day A	Zn: μg/day B
0-1	0.90	0.23	2.9	160	0.01	0.01	0.03	7.0
1-2	0.48	0.11	130	41		<0.01		NM
2-3	0.40	<0.10	26	NM		<0.01		NM
3-5	0.22	.11	15	32		<0.01		NM
5-7	0.20	<0.10	13	NM	0.02		1.3	
7-14	0.10	0.10	6.6	27	<0.01		NM	
14-21	0.10		6.4		<0.01		NM	

A = Head pressure = 4 inches
B = Head pressure = 11.5 inches
NM = Not Meaningful and therefore not calculated
Blanks indicate test not run
*This is a minimum value because the leachate collector overflowed

Table 17

Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Deionized Water at Different Head Pressures

Plant 6

Day	pH A	pH B	Conductivity (µmhos/cm) A	Conductivity (µmhos/cm) B	Amount Collected (g/day) A	Amount Collected (g/day) B	Cr, EP = 25.4 mg/l: mg/l A	Cr mg/l B	Cr µg/day A	Cr µg/day B	Pb, EP = 0.001 mg/l: mg/l A	Pb mg/l B	Pb µg/day A	Pb µg/day B
0-1	8.2	9.11	290	750	102	900	<0.05	0.10	NM	90	<0.001	<0.001	NM	NM
1-2	8.6	7.65	170	175	118	1093	<0.05	0.05	NM	55		<0.001		NM
2-3	8.0	7.80	125	112	117	993	<0.05	0.05	NM	50		0.003		3.0
3-5	8.4	8.60	73	99	114	1008	<0.05	0.06	NM	61		<0.001		NM
5-7	7.4	9.65	210	80	116	1040	<0.05	0.06	NM	62	<0.001	0.003	NM	3.1
7-14	8.1	9.50	204	60	115	1208	<0.05	0.10	NM	120	<0.001	<0.001	NM	NM
14-21	7.9		145		118		<0.05		NM		<0.001		NM	

Day	Cu, EP = 0.02 mg/l: mg/l A	Cu mg/l B	Cu µg/day A	Cu µg/day B	Mn, EP = 9.6 mg/l: mg/l A	Mn mg/l B	Mn µg/day A	Mn µg/day B	Ni, EP = 79 mg/l: mg/l A	Ni mg/l B	Ni µg/day A	Ni µg/day B
0-1	0.02	<0.01	2.0	NM	<0.1	<0.1	NM	NM	0.20	<0.10	20	NM
1-2		<0.01		NM	<0.1	<0.1	NM	NM	0.12	<0.10	14	NM
2-3		<0.01		NM	<0.1	<0.1	NM	NM	<0.10	<0.10	NM	NM
3-5		0.02		20		<0.1		NM		<0.10		NM
5-7	0.02	<0.01	2.3	NM	<0.1		NM		<0.10		NM	
7-14	<0.01	<0.01	NM	NM	<0.1		NM					
14-21	<0.01		NM		<0.1		NM					

Day	Zn, EP = 0.33 mg/l: mg/l A	Zn mg/l B	Zn µg/day A	Zn µg/day B
0-1	0.02	<0.01	2	NM
1-2		<0.01		NM
2-3		<0.01		NM
3-5		<0.01		NM
5-7	0.01		1.2	
7-14	<0.01		NM	
14-21				

A = Head pressure = 4 inches
B = Head pressure = 11.5 inches
NM = Not Meaningful and therefore not calculated
Blanks indicate test not run
*This is a minimum value because the leachate collector overflowed

Table 17

Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Deionized Water at Different Head Pressures

Plant 8

Day	pH		Conductivity (μmhos/cm)		Amount Collected (g/day)		Ag, EP = 0.02 mg/l				Cr, EP = 400 mg/l			
							mg/l		μg/day		mg/l		μg/day	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B
0-1	8.4	6.70	2280	775	246	1370	0.02	0.02	4.9	27	<0.05	<0.05	NM	NM
1-2	8.4	8.60	2290	980	277	1660		<0.01		NM	<0.05	<0.05	NM	NM
2-3	8.4	8.30	2040	720	274	1524		<0.01		NM	<0.05	<0.05	NM	NM
3-5	8.4	6.40	1700	700	359	1394		<0.01		NM	<0.05	<0.05	NM	NM
5-7	8.6	8.65	1110	570	540	1578	<0.01		NM		<0.05		NM	
7-14	8.3	8.30	550	313	601	941	<0.01		NM		<0.05		NM	
14-21	8.2		115		1301		<0.01		NM		<0.05		NM	

Day	Hg, EP = 0.045 mg/l				Pb, EP = 0.032 mg/l				Cu, EP = 29.9 mg/l			
	mg/l		μg/day		mg/l		μg/day		mg/l		μg/day	
	A	B	A	B	A	B	A	B	A	B	A	B
0-1	0.0128	0.0019	3.1	2.6	<0.001	<0.001	NM	NM	0.47	0.14	116	192
1-2	0.0134	0.0027	3.7	4.5		<0.001		NM	0.19	0.05	53	83
2-3	0.0020	0.0019	.55	2.9	<0.001	<0.001	NM	NM	0.31	0.02	85	30
3-5	0.0015	0.0012	.53	1.7		<0.001		NM	0.07	0.02	25	28
5-7	0.0008	0.0010	.43	1.6	<0.001		NM		0.05	0.02	27	32
7-14	0.0006	<.0005	.36	NM	<0.001		NM		0.03	0.02	18	19
14-21	<.0005		NM		<0.001		NM		<0.01		<13	

Day	Mn, EP = 1.52 mg/l				Ni, EP = 375 mg/l				Zn, EP = 0.14 mg/l			
	mg/l		μg/day		mg/l		μg/day		mg/l		μg/day	
	A	B	A	B	A	B	A	B	A	B	A	B
0-1	<0.10	<0.10	NM	NM	0.12	0.14	30	192	<0.01	<0.01	NM	NM
1-2		<0.10		NM	0.12	<0.10	33	NM	<0.01	<0.01	NM	NM
2-3	<0.10	<0.10	NM	NM	<0.10	<0.10	NM	NM	0.01	<0.01	2.7	NM
3-5		<0.10		NM		<0.10		NM		<0.01		NM
5-7	<0.10		NM		<0.10		NM		<0.01		NM	
7-14	<0.10		NM									
14-21	<0.10		NM									

A = Head pressure = 4 inches
B = Head pressure = 11.5 inches
NM = Not Meaningful and therefore not calculated
Blanks indicate test not run
*This is a minimum value because the leachate collector overflowed

Table 17

Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Deionized Water at Different Head Pressures

Plant 11

Day	pH A	pH B	Conductivity (µmhos/cm) A	Conductivity (µmhos/cm) B	Amount Collected (g/day) A	Amount Collected (g/day) B	Ag, EP = <0.01 mg/l mg/l A	Ag mg/l B	Ag µg/day A	Ag µg/day B	As, EP = 0.035 mg/l mg/l A	As mg/l B	As µg/day A	As µg/day B
0-1	9.3	9.55	62000	23500	225	516	0.08	0.04	18	21	0.24	0.025	54	13
1-2	9.4	9.25	10000	450	477	932	0.02	<0.01	10	NM	0.008	<0.001	3.8	NM
2-3	9.4	8.40	1120	109	562	890	<0.01	<0.01	NM	NM	0.002		1.1	
3-5	8.7	7.40	210	80	620	867	<9.01	<0.01	NM	NM	<0.001	<0.001	NM	NM
5-7	8.4	7.95	98	63	661	866	<0.01		NM		<0.001		NM	
7-14	7.9	7.80	60	45	598	553	<0.01		NM		<0.001		NM	
14-21	7.8		48		443		<0.01		NM		<0.001		NM	

Day	Cr, EP = 4.22 mg/l mg/l A	Cr mg/l B	Cr µg/day A	Cr µg/day B	Pb, EP = 0.004 mg/l mg/l A	Pb mg/l B	Pb µg/day A	Pb µg/day B	Cu, EP = 0.24 mg/l mg/l A	Cu mg/l B	Cu µg/day A	Cu µg/day B
0-1	<0.05	0.05	NM	26	<0.001	0.009	NM	4.6	0.29	0.14	65	72
1-2	0.05	<0.05	24	NM		<0.001		NM	0.06	<0.01	29	NM
2-3	<0.05	<0.05	NM	NM		<0.001		NM	0.03	<0.01	17	NM
3-5	<0.05	<0.05	NM	NM		<0.001		NM	0.01	<0.01	6	NM
5-7	<0.05		NM		<0.001		NM		<0.01		NM	
7-14	<0.05		NM		<0.001		NM		<0.01		NM	
14-21	<0.05		NM		<0.001		NM		<0.01		NM	

Day	Mn, EP = 1.71 mg/l mg/l A	Mn mg/l B	Mn µg/day A	Mn µg/day B	Ni, EP = 71.0 mg/l mg/l A	Ni mg/l B	Ni µg/day A	Ni µg/day B	Zn, EP = 941 mg/l mg/l A	Zn mg/l B	Zn µg/day A	Zn µg/day B
0-1	<0.10	<0.10	NM	NM	0.45	0.22	101	114	1.29	0.18	290	93
1-2		<0.10		NM	<0.10	<0.10	NM	NM	0.06	<0.01	29	NM
2-3		<0.10		NM	<0.10	<0.10	NM	NM	0.04	<0.01	22	NM
3-5		<0.10		NM		<0.10		NM	0.01	0.02	6	17
5-7	<0.10		NM		<0.10		NM		0.02	0.02	13	17
7-14	<0.10		NM						0.03	<0.01	18	NM
14-21	<0.10		NM									

A = Head pressure = 4 inches
B = Head pressure = 11.5 inches
NM = Not Meaningful and therefore not calculated
Blanks indicate test not run
*This is a minimum value because the leachate collector overflowed

Table 17

Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Deionized Water at Different Head Pressures

Plant 11 - Duplicate

Day	pH A	pH B	Conductivity (µmhos/cm) A	Conductivity (µmhos/cm) B	Amount Collected (g/day) A	Amount Collected (g/day) B	Ag, EP = <0.01 mg/l: mg/l A	Ag: mg/l B	Ag: µg/day A	Ag: µg/day B	As, EP = 0.035 mg/l: mg/l A	As: mg/l B	As: µg/day A	As: µg/day B
0-1	9.3	9.6	69000	16800	234	946	0.09	0.04	21	38	0.350	0.032	82	30
1-2	9.45	8.6	9000	220	530	1213	<0.01	<0.01	NM	NM	0.007	<0.001	4	NM
2-3	9.2	6.4	550	81	1080	1068	<0.01	<0.01	NM	NM	<0.001		NM	
3-5	8.5	6.3	110	65	1071	1070	<0.01	<0.01	NM	NM	<0.001	<0.001	NM	NM
5-7	8.2	8.0	80	51	1090	1099	<0.01		NM		<0.001		NM	
7-14	7.5	7.65	52	37	1056	878	<0.01		NM		<0.001		NM	
14-21	7.6		38		934		<0.01		NM		<0.001		NM	

Day	Cr, EP = 4.22 mg/l: mg/l A	Cr: mg/l B	Cr: µg/day A	Cr: µg/day B	Pb, EP = 0.004 mg/l: mg/l A	Pb: mg/l B	Pb: µg/day A	Pb: µg/day B	Cu, EP = 0.24 mg/l: mg/l A	Cu: mg/l B	Cu: µg/day A	Cu: µg/day B
0-1	0.18	0.05	42	47	0.006	0.009	1.4	8	0.34	0.14	80	132
1-2	<0.05	<0.05	NM	NM		<0.001		NM	0.45	<0.01	238	NM
2-3	<0.05	<0.05	NM	NM		<0.001		NM	0.01	<0.01	11	NM
3-5	<0.05	<0.05	NM	NM		<0.001		NM	0.02	<0.01	21	NM
5-7	<0.05		NM		0.002		2.2		<0.01		NM	
7-14	<0.05		NM		<0.001		NM		<0.01		NM	
14-21	<0.05		NM		<0.001		NM		<0.01		NM	

Day	Mn, EP = 1.71 mg/l: mg/l A	Mn: mg/l B	Mn: µg/day A	Mn: µg/day B	Ni, EP = 71.0 mg/l: mg/l A	Ni: mg/l B	Ni: µg/day A	Ni: µg/day B	Zn, EP = 941 mg/l: mg/l A	Zn: mg/l B	Zn: µg/day A	Zn: µg/day B
0-1	<0.10	<0.10	NM	NM	0.35	0.16	82	151	0.90	0.27	211	255
1-2		<0.10		NM	<0.10	<0.10	NM	NM	0.06	<0.01	32	NM
2-3		<0.10		NM	<0.10	<0.10	NM	NM	0.01	<0.01	11	NM
3-5		<0.10		NM		<0.10		NM	0.02	0.01	21	11
5-7	<0.10		NM		<0.10		NM		0.02	0.02	22	22
7-14	<0.10		NM						0.03	0.01	32	9
14-21	<0.10		NM						0.01		9	

A = Head pressure = 4 inches
B = Head pressure = 11.5 inches
NM = Not Meaningful and therefore not calculated
Blanks indicate test not run
*This is a minimum value because the leachate collector overflowed

Table 17

Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Deionized Water at Different Head Pressures

Plant 12

Day	pH A	pH B	Conductivity (μmhos/cm) A	Conductivity (μmhos/cm) B	Amount Collected (g/day) A	Amount Collected (g/day) B	Ag, EP = 0.03 mg/l: mg/l A	Ag, EP = 0.03 mg/l: mg/l B	Ag, EP = 0.03 mg/l: μg/day A	Ag, EP = 0.03 mg/l: μg/day B	Cd, EP = 268 mg/l: mg/l A	Cd, EP = 268 mg/l: mg/l B	Cd, EP = 268 mg/l: μg/day A	Cd, EP = 268 mg/l: μg/day B
0-1	8.4	7.3	900	680	248	700	0.04	0.02	10	14	0.24	0.18	60	126
1-2	8.35	8.2	600	318	206	771		<0.01		NM	0.18	0.12	37	93
2-3	8.2	8.19	455	265	343	832		<0.01		NM	0.16	0.09	55	75
3-5	8.2		340	225	339	770		<0.01		NM	0.12	0.08	41	62
5-7	8.2	8.1	270	200	352	662	<0.01		NM		0.11	0.08	39	53
7-14	8.0	7.5	230	180	271	488*	<0.01		NM		0.09	0.06	24	>29
14-21	8.0		215		231		<0.01		NM		0.08		19	

Day	Cr, EP = 4.85 mg/l: mg/l A	Cr, EP = 4.85 mg/l: mg/l B	Cr, EP = 4.85 mg/l: μg/day A	Cr, EP = 4.85 mg/l: μg/day B	Pb, EP = 0.031 mg/l: mg/l A	Pb, EP = 0.031 mg/l: mg/l B	Pb, EP = 0.031 mg/l: μg/day A	Pb, EP = 0.031 mg/l: μg/day B	Cu, EP = 19.2 mg/l: mg/l A	Cu, EP = 19.2 mg/l: mg/l B	Cu, EP = 19.2 mg/l: μg/day A	Cu, EP = 19.2 mg/l: μg/day B
0-1	0.34	0.22	84	154	<0.001	<0.001	NM	NM	0.86	0.12	213	84
1-2	0.21	0.10	43	77		<0.001		NM		0.07		54
2-3	0.15	0.08	51	67		<0.001		NM		0.05		42
3-5	0.12	0.05	41	39		0.001		0.8	0.16	0.05	54	39
5-7	<0.05	0.05	NM	33	<0.001	<0.001	NM	NM	0.14	0.04	49	26
7-14	0.07	<0.05	19	NM	<0.001	<0.001	NM	NM	0.13	0.03	35	>15
14-21	0.05		18		<0.001		NM		0.08		19	

Day	Mn, EP = 23.8 mg/l: mg/l A	Mn, EP = 23.8 mg/l: mg/l B	Mn, EP = 23.8 mg/l: μg/day A	Mn, EP = 23.8 mg/l: μg/day B	Ni, EP = 14.8 mg/l: mg/l A	Ni, EP = 14.8 mg/l: mg/l B	Ni, EP = 14.8 mg/l: μg/day A	Ni, EP = 14.8 mg/l: μg/day B
0-1	<0.10	<0.10	NM	NM	<0.10	<0.10	NM	NM
1-2		<0.10		NM		<0.10		NM
2-3		<0.10		NM		<0.10		NM
3-5		<0.10		NM		<0.10		NM
5-7	<0.10		NM		<0.10		NM	
7-14	<0.10		NM					
14-21	<0.10		NM					

A = Head pressure = 4 inches
B = Head pressure = 11.5 inches
NM = Not Meaningful and therefore not calculated
Blanks indicate test not run
*This is a minimum value because the leachate collector overflowed

after 14 days at an equivalent rainfall of 24 inches per day, the pH of Plant 4's leachate fell only from 9.32 to 8.95, which is an indication of the tremendous buffering power present. In an area of the country where 40 inches of rain per year is normal, this would amount to 8.4 years of average rainfall. Furthermore, in a properly clay-covered landfill where clay permeabilities are on the order of 10^{-6} cm/sec, this amount of water would require many times this period to percolate through the cover layer to reach the sludge, especially since proper design would avoid significant head pressures. Evaporation and runoff would greatly increase this time period. The lowest final pH observed in any of the tests was 7.6. Phase I testing has shown that solubility of the toxic metals is strongly dependent upon pH, with generally lower solubilities at higher pH's. Dynamic testing indicates that the pH of the electroplating sludges will remain elevated even after copious amounts of deionized water have passed through the sludge.

The conductivity of the leachate water was monitored to yield a readily obtainable indicator of the ion content of the water, and thus a measure of the leaching activity. As expected, the conductivity invariably started high and dropped off with time as the testing progressed. The initial high levels were expected because this is when the supernatent and interstitial waters would be flushed out with their high content of soluble materials. After this material is flushed out, any ion content in the leachate waters must come from the dissolution of material in the sludge itself. A large, rapid drop in conductivity (as in

Plant 11 where conductivity dropped by a factor of over 1000) indicates that very little material is actually being leached after the initial flushing. The actual amounts of metals being leached correlates well with the conductivity, as indicated in Table 17 which shows metal concentrations and quantities decreasing with decreasing conductivities. Subsequent testing showed that the bulk of the conductivity cannot be attributed to metals but rather to the other ions present in the water. Tests on metal solutions in the 10 mg/l concentration range showed conductivities of approximately 20 mhos/cm. In every dynamic test but one, the conductivity eventually decreased to a value substantially lower than the initial value.

The levels of metals detected in the leachate are presented in Table 17. What is most striking about these levels is how low they are. When the volume of leaching water is considered, the mass of the metals leached per day is very low, as demonstrated by there being only three individual results with metal concentrations greater than 1.0 mg/l (two of which are not on EPA's hazardous list) and with the exception of one barium result showing 4 mg/day being leached, no other metal leached at a rate as high as 0.7 mg/day at any time. For reference, the sludges leached weighed approximately 200 g (wet weight). Another striking observation from Table 17 is that the results from the EPA's Extraction Procedure (EP) have little correspondence to what was leached from the sludge during dynamic testing. For example, zinc from Plant 4 had an EP value of 648 mg/l but Zn was virtually undetected during dynamic leaching with deionized

water as was chromium from Plant 8, which had an EP level of 400 mg/l. Although more testing is required, these test results support the conclusion that electroplating sludges in a segregated hydroxide environment will not leach significant quantities of toxic metals. The vast majority of metals analyzed for during the dynamic testing were either present below detectable levels or very close to the detectable levels after the first day.

The concentration of metals varied significantly between tests run on the same sludge at two different heads. This was due primarily to the increased volume of leaching water at the higher head pressure. However, the actual quantities of metal leached were essentially the same for each set of tests conducted. Note specifically Cr at Plant 4, Ni at Plant 5, Hg and Cu at Plant 8, and Cd and Cr at Plant 12 in Table 17. Unfortunately there were not enough metals leached in most of the other tests to further support this observation. One explanation of this phenomenon is that the leaching rate is controlled at least partially by the diffusion of metal ions through the sludge particle and the particle/liquid interface, or, as it is more commonly called, the boundary layer. For any leaching to occur after the initial flushing is complete, the metals must migrate through the sludge particle (which may be the rate determining factor), then through the boundary layer and finally into the water stream. If boundary layer dissusion predominates, no matter how much leaching water passes by the sludge particle, the rate of metal ion movement (flux) through the boundary layer would remain essentially constant. Dynamic test results show leaching rates dropping off

with time, which supports the concept of ion migration through the sludge particle as the real rate-determining factor. As more metal ions are leached, the remaining ions have a continually longer route to the surface of the particle, resulting in lower leaching rates. High EP results for a particular metal ion demonstrate that large quantities of that metal are present in the sludge. Coupling this with the low metal quantities leached during dynamic testing indicates that the diffusion rate of heavy metals is very low when leached with deionized water.

4.3 DYNAMIC TESTING WITH ACIDIFIED WATER

Dynamic testing with deionized water demonstrated that the electroplating sludges tested would leach very low metal levels when segregated and protected from an acid environment. Even in a real world segregated hydroxide landfill, however, the sludges could not be assured of an acid-free environment in some areas of the country due to the possibility of acid-rain. Acid-rain has been cited in the news frequently, for damaging foliage and lakes, along with causing other maladies. Dynamic testing was conducted with an acidified water as the leaching water to determine its effect on unprotected electroplating sludges. For this experiment, CO_2 was bubbled through the main water supply reservoirs to simulate the CO_2 which would be absorbed by rain water from the atmosphere. Sulfuric acid was added to yield a $SO_4^=$ concentration of 3 mg/l and a pH of 4.21. These levels are consistent with levels found in the literature for acid-rain. Table 18 shows the actual pH and conductivity of the acidified water as it was monitored during the testing. It

Table 18

Conductivity & pH of Acidified Water

Used for Leaching in Dynamic Testing

Day	Conductivity (μmhos/cm)	pH
0-1	40	4.00
1-2	25	4.00
2-3	20	4.10
3-5	23	4.10
5-7	25	4.15
7-14	20	4.20

would be very difficult to simulate an actual acid rainfall as the acidity is constantly changing in such a rain--starting out at its strongest level and gradually becoming weaker. To be conservative and to test worst-case conditions, the testing was performed at the most acidic conditions found in acid-rain for the entire length of the experiment.

The head pressure for the acidified water tests was 4 inches of water and daily flow rates of leaching water remained approximately constant. The flows were generally higher during the acidified water testing than during the DIW testing at the same head pressure which may be explained by the variability inherent in the seating of the sludge in the apparatus.

Flow rates ranged from approximately 500 to 2000 g of leachate collected per day (the equivalent of 2.4 to 9.7 inches of rain/day). As discussed earlier this represents many years of rain contact with sludge. The pH levels in the leachate ranged from 6.0 to 9.0 but were in the 6.1 to 7.5 range for most of the test period. The largest drop in pH for any single test was from 9.0 to 6.3. The ranges in pH are greater than those observed with DI water leaching, as would be expected.

Conductivity of the leachate followed the same general pattern with acidified water as with DI water testing in that they both started off at relatively high levels and decreased over the test period. The major difference was that with DI water the conductivity dropped off rapidly while with simulated acidified water the conductivity dropped sharply after the first day but only

slowly after that. Conductivity seemed to have little correlation with the quantity of metals leached, as shown in Table 19.

The acidified leaching water had rather dramatic effects on the quantities of specific metals leached. This effect was most notable on Ni, Zn, Cd, Mn, Cu, and Cr, in that order, where substantially higher metal quantities were leached. These higher leaching levels were expected due to the lower pH of the leaching water. Unlike the dynamic tests with DI water, where metal quantities in the leachate started high and dropped quickly, the quantities leached with acidified water tended to peak on the third day before dropping off slowly to negligible quantities. A few metals did not behave in this manner, but leached as they did when subjected to DI water tests, that is with the high leaching rate on the first day dropping off almost immediately. This may be because these metals were primarily in the supernatant and interstitial waters and not in the sludge--which means that there would be little to leach after the initial flushing.

Leaching rates climbing to a maximum and then falling would agree with the boundary-layer-diffusion rate-limiting theory suggested in the previous section. The acidified water effect on the sludge particles would be initially minimized by the presence of excess lime, which is more readily attacked by the acid. Table 19 indicates that in general the metals were still leached in significantly higher quantities on the first two days than was occurring with the DI water tests for the same time period. After a couple of days of flushing, the sludge particles lose

Table 19
Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Acidified Water

Plant 5

Day	pH	Conductivity (μmhos/cm)	Amount Collected (g/day)	As		Cr		Pb		Cu		Mn	
				mg/l	μg/day	mg/l	μg/day	mg/l	μg/day	mg/l	μg/day	mg/l	μg/day
0-1	7.5	1750	518	0.015	7.8	<0.02	NM	0.001	0.6	<0.01	<5.2	0.04	20.7
1-2	7.33	830	568	<0.001	NM	<0.02	NM	<0.001	NM	<0.01	<5.6	0.05	28.4
2-3	7.10	600	601	<0.001	NM	<0.02	NM	<0.001	NM	0.04	24.0	0.08	48.1
3-5	7.10	389	512							0.01	5.1	0.10	51.2
5-7	6.8	227	470							<0.01	<4.7	0.06	28.2
7-14	7.2	136	422										

Day	Ni		Zn	
	mg/l	μg/day	mg/l	μg/day
0-1	1.00	518	0.02	10.4
1-2	1.85	1051	0.02	11.4
2-3	2.45	1472	0.02	12.0
3-5	2.50	1281	0.01	5.1
5-7	1.85	868	<0.01	NM
7-14	1.54	650		

NM = Not Meaningful and therefore not calculated

*This is a minimum value because the leachate collector overflowed

Blanks indicate tests not run

Table 19
Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Acidified Water

Plant 6

Day	pH	Conductivity (μmhos/cm)	Amount Collected (g/day)	Cr		Pb		Cu		Mn		Ni	
				mg/l	μgm/day	mg/l	μgm/day	mg/l	μgm/day	mg/l	μgm/day	mg/l	μgm/day
0-1	8.70	1000	698	0.24	168	<0.001	NM	<0.01	NM	<0.02	NM	<0.10	NM
1-2	7.45	383	995	0.04	40	<0.001	NM	<0.01	NM	<0.02	NM	0.11	109
2-3	6.80	322	1121	0.03	34	<0.001	NM	<0.01	NM	0.03	34	0.25	280
3-5	7.50	240	756							0.02	15	0.10	76
5-7	7.10	135	868									0.10	87
7-14	6.90	93	784*										

Day	Zn	
	mg/l	μgm/day
0-1	0.01	7
1-2	0.02	20
2-3	0.02	22
3-5	0.02	15
5-7	<0.01	NM
7-14		

NM = Not Meaningful and therefore not calculated

*This is a minimum value because the leachate collector overflowed

Blanks indicate tests not run

Table 19
Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Acidified Water

Plant 8

Day	pH	Conductivity (μmhos/cm)	Amount Collected (g/day)	Ag		Cr		Hg		Pb		Cu	
				mg/l	μg/day	mg/l	μg/day	mg/l	μg/day	mg/l	μg/day	mg/l	μg/day
0-1	7.00	1500	1532	0.01	15	0.10	153	0.0159	24	<0.001	NM	0.22	337
1-2	7.00	800	2229	<0.01	NM	0.10	223	0.0075	17	0.001	2.2	0.29	646
2-3	6.40	670	2496	<0.01	NM	0.08	200	0.0059	15	0.001	2.5	0.38	948
3-5	6.00	480	2572			0.08	206	0.0063	16			0.34	875
5-7	6.10	305	2516			0.02	50	0.0009	2.3			0.09	226
7-14	6.40	140	3204*			<0.02	NM					0.11	>352

Day	Mn		Ni		Zn	
	mg/l	μg/day	mg/l	μg/day	mg/l	μg/day
0-1	0.03	46	2.40	3677	0.02	31
1-2	0.03	67	3.40	7579	0.03	67
2-3	0.03	75	3.35	8362	0.04	100
3-5	0.03	77	2.80	7203	0.01	26
5-7	<0.02	50	0.75	1887	0.02	50
7-14			0.68	>2179		

*This is a minimum value because the leachate collector overflowed

Table 19
Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Acidified Water

Plant 11

Day	pH	Conductivity (μmhos/cm)	Amount Collected (g/day)	Ag		As		Cr		Pb		Cu	
				mg/l	μgm/day	mg/l	μgm/day	mg/l	μgm/day	mg/l	μgm/day	mg/l	μgm/day
0-1	9.00	12300	893	0.04	36	0.170	152	0.04	36	0.003	2.7	0.11	98
1-2	7.75	1300	1084	<0.01	NM	0.001	1.1	0.03	33	0.002	2.2	0.02	22
2-3	6.90	360	1234	<0.01	NM	<0.02	NM	<0.02	NM	<0.001	NM	<0.01	NM
3-5	6.90	160	1152									0.01	12
5-7	6.70	108	1162									<0.01	NM
7-14	6.30	82	991										

Day	Mn		Ni		Zn	
	mg/l	μgm/day	mg/l	μgm/day	mg/l	μgm/day
0-1	0.08	71	0.80	714	6.00	5358
1-2	0.14	152	2.05	2222	17.25	18699
2-3	0.13	160	2.10	2591	18.00	22212
3-5	0.08	92	1.30	1498	12.50	14400
5-7	0.05	58	0.70	814	6.50	7556
7-14	0.05	50	0.74	734	5.20	5155

*This is a minimum value because the leachate collector overflowed

Table 19
Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Acidified Water

Plant 11 Duplicate

Day	pH	Conductivity (μmhos/cm)	Amount Collected (g/day)	Ag		As		Cr		Pb		Cu	
				mg/l	μgm/day	mg/l	μgm/day	mg/l	μgm/day	mg/l	μgm/day	mg/l	μgm/day
0-1	8.60	11700	885	0.04	35	0.140	124	0.04	35	0.002	1.8	0.12	106
1-2	7.25	1850	981	<0.01	NM	0.002	2	0.03	29	<0.001	NM	0.01	10
2-3	6.48	520	1107	<0.01	NM	<0.001	NM	0.02	22	<0.001	NM	<0.01	NM
3-5	6.90	225	1107									0.01	11
5-7	6.90	138	1002									<0.01	NM
7-14	6.30	102	846										

Day	Mn		Ni		Zn	
	mg/l	μgm/day	mg/l	μgm/day	mg/l	μgm/day
0-1	0.13	115	1.60	1416	11.50	10178
1-2	0.22	216	3.35	3286	24.50	24034
2-3	0.23	255	4.10	4539	35.00	38745
3-5	0.12	133	2.05	2269	16.30	18044
5-7	0.07	70	1.05	1053	8.00	8020
7-14	0.06	51	1.18	998	7.80	6598

*This is a minimum value because the leachate collector overflowed

Table 19
Leachate Characteristics from Dynamic Testing of Electroplating Sludge with Acidified Water

Plant 12

Day	pH	Conductivity (μmhos/cm)	Amount Collected (g/day)	Ag		Cd		Cr		Pb		Cu	
				mg/l	mg	mg/l	mg	mg/l	mg	mg/l	mg	mg/l	mg
0-1	7.20	800	1019	0.01	10	5.55	5655	0.15	153	<0.001	NM	0.28	285
1-2	6.93	570	1120	<0.01	NM	7.95	8904	0.06	67	<0.001	NM	0.39	437
2-3	6.55	429	1330	<0.01	NM	8.15	10840	0.04	53	<0.001	NM	0.52	692
3-5	7.10	273	1244*			3.28	>4080	<0.02	NM			0.18	>224
5-7	7.15	170	1562			1.80	2812	<0.02	NM			0.10	156
7-14	6.10	140	1441			1.85	2667					0.12	173

Day	Mn		Ni		Zn	
	mg/l	mg	mg/l	mg	mg/l	mg
0-1	0.15	153	0.50	509	3.00	3057
1-2	0.18	202	0.80	896	5.50	6160
2-3	0.16	213	0.95	1264	6.25	8312
3-5	0.06	>35	0.35	>435	2.25	>2799
5-7	0.02	31	0.10	156	1.00	1562
7-14			0.15	216	1.00	1441

*This is a minimum value because the leachate collector overflowed

some of the protection provided by excess lime and metal leaching reaches its maximum rate. After a short period of heavy leaching, the metal ions must travel increasingly longer routes to the surface of the sludge particle (the sludge/water interface). This then becomes rate limiting and the leaching rate begins to drop off substantially.

The above is only one of several mechanisms that can be postulated. Whatever the actual mechanism is, it is obvious that an acid-rain could result in higher leaching rates of heavy metals from unprotected electroplating sludges. Of the metals currently proposed as being potentially hazardous under RCRA, only cadmium leaching rates appear to be significantly affected. The fact that the quantities of acidified water used in these experiments plus the high constant acidity represent many decades of exposure in a properly designed landfill to acid rain should be considered before drawing conclusions as to the potential environmental impact of these sludges. Possible preventative measures for a segregated landfill in an area of the country subject to acid-rain include covering with limestone or an impermeable barrier to prevent percolation. An impermeable barrier would prevent the sludge from coming in contact with rainwater and thus obviate the problem. As a final note on this subject, it is important to remember that acid-rain is regional and is not a problem for most areas of the United States.

SECTION 5

DISCUSSION OF PHASE III EXPERIMENTS

While analytical laboratory work under controlled conditions is a necessary part of the characterization of the leachability of electroplating sludges, it is highly desirable to evaluate a real world sludge disposal site to verify or calibrate conclusions drawn from the laboratory work. In light of this desire, one of the 12 plants initially sampled during this study, Plant 11, was selected because it had on-site landfill disposal of electroplating sludges. There were actually two disposal sites at the plant.

The first landfill site is merely a bulldozed hole in the ground with the displaced dirt forming a retaining wall between the landfill and a small creek approximately 50 feet away. This site had not been used in over 6 years, had thick cattail growth, and was teeming with bullfrog tadpoles. This pond had been drained of 54 inches of sludge and water just prior to the visit to allow sludge sampling. A sludge/soil interface was visually detectable. Samples were taken from: the sludge layer; 11 inches below the interface; and 17 inches below the interface.

The second landfill site has been active for the last 6 years. It was constructed by scraping away topsoil to a layer of limestone rock and using the displaced soil for retaining walls. A layer of limestone was then spread over the inner retaining walls and the bottom of the landfill. The landfill area was surrounded

by a drainage ditch which collected any runoff or leachate from the landfill. Samples were taken: at the surface of the sludge; directly below that sample at the limestone rock layer; and at the limestone rock layer from an older portion of the landfill. A water sample was taken from the drainage ditch and a background soil sample was taken from the vicinity of the older landfill site.

All of the solid samples were subjected to the EPA Extraction Procedure (EP) to generate data which could be compared to Phase I and II results, and the water sample was analyzed for its concentration of metal ions. The results are presented in Table 20 and illustrate many interesting points about the sludge generated by this plant. After at least 6 years of unprotected exposure to the elements, the sludge in the older landfill showed typically higher EP results than did the soil levels below it. Except for Mn, the soil samples below the old landfill are not substantially different from the background soil sample taken. To determine if the heavy metals had been merely washed away over the years, sludge samples were tested for their total Cr content. These tests (also shown in Table 20) proved that a substantial quantity of Cr still remained in the sludge and that even the aggressive nature of the EP could only remove a small portion of it. Soil from 17 inches below the sludge interface yielded EP results that were indistinguishable from the background soil.

The newer landfill had substantially higher EP results for Cr and three of the non-hazardous metals (i.e., Cu, Ni and Zn). The Cr

Table 20

PHASE III FIELD TESTS

Characterization of Samples Taken from Segregated Landfills

Plant Site 11

Sample Description	EP Results in mg/l							Cr Concentration in Sample
	Ag	As	Cu	Mn	Ni	Zn	Cr	(µg/Kg or ppb)
NEW LANDFILL								
Surface Sludge	0.02	0.006	0.04	2.60	45.0	831	3.58	58,500
Subsurface Sludge	0.01	0.005	7.24	2.55	50.6	1110	4.37	
Older Subsurface Sludge	<0.01	0.002	0.34	2.90	28.1	360	3.35	98,200
OLD LANDFILL								
Sludge Layer	0.19	0.002	<0.01	11.3	1.73	0.08	0.09	2,100.0
11" Below Sludge	0.03	0.022	0.03	1.62	<0.10	0.12	<0.05	450.0
17" Below Sludge	0.02	0.011	<0.01	2.08	<0.10	0.08	<0.05	41.3
BACKGROUND SOIL	<0.01	0.004	<0.01	<0.10	<0.10	0.06	<0.05	32.4
NEW LANDFILL LEACHATE ANALYSIS	0.01	0.004	0.02	2.18	<0.10	0.04	<0.05	

Note: Samples were also analyzed for Ba, Cd, and Se, but all results were below detection levels. Blanks indicate tests not run.

concentration in the new landfill sludge was 40 to 50 times higher than in the older landfill, however, once again this Cr is tightly held by the sludge, as evidenced by the EP results and the undetectable Cr level in the leachate from the newer landfill. Note that production changes over the years make conclusions drawn from the comparison of sludge samples from one site to the other of dubious validity. Perhaps the most striking test result of this phase of the study is that the actual leachate from a landfill which contained electroplating wastewater treatment sludges having very high EP results for five metals would readily pass currently proposed drinking water standards, except for Mn.

Testing conducted during this final phase of the study indicates that in spite of very high metal concentrations in the sludges, that these metals are not easily leached out of the sludges in a segregated landfill environment. This indication is supported by EP results on the sludges and underlying soils and is further borne out by the low metal concentrations in the leachate from the landfill. In this respect the Phase III field test results have verified the Phase II dynamic tests results which indicated that the sludge in a segregated environment would leach heavy metals at a very low rate after the initial flushing of the supernatant and interstitial waters.

APPENDIX A

ANALYTICAL QUALITY CONTROL

All sample preparation and analysis were performed in accordance with those procedures set forth by the EPA ("Methods for Chemical Analysis of Water and Wastes," 1974). This includes the use of high quality deionized water for samples, blanks and standards; adequate glassware cleaning procedures, the use of Ultrex™ acids for wet digestion, and subsequent analysis via flame, cold vapor (Hg), and furnace AAS.

Ten percent of all samples submitted for analysis were analyzed in duplicate; an additional 10 percent were spiked. These results were used to compute precision and accuracy data which was compared to previously generated analytical control data for each element analyzed. Trouble-shooting was performed if results did not fall within acceptable limits.

	Detection Limit (mg/l)		
Ag	0.01	As	0.001
Ba	1.00	Cd	0.01
Cr	0.05	Cu	0.01
Mn	0.10	Ni	0.10
Pb	0.001	Se	0.005
Zn	0.01	Hg	0.0005

APPENDIX A (Continued)

Glassware Cleaning Procedure

1. Remove all loose dirt and foreign material with several tap water rinses.

2. Remove visible solids by scrubbing with detergent and hot water. Rinse with hot water three times.

3. Rinse with "No-Chromex" (concentrated H_2SO_4 saturated with ammonium persulfate). Rinse with cold tap water.

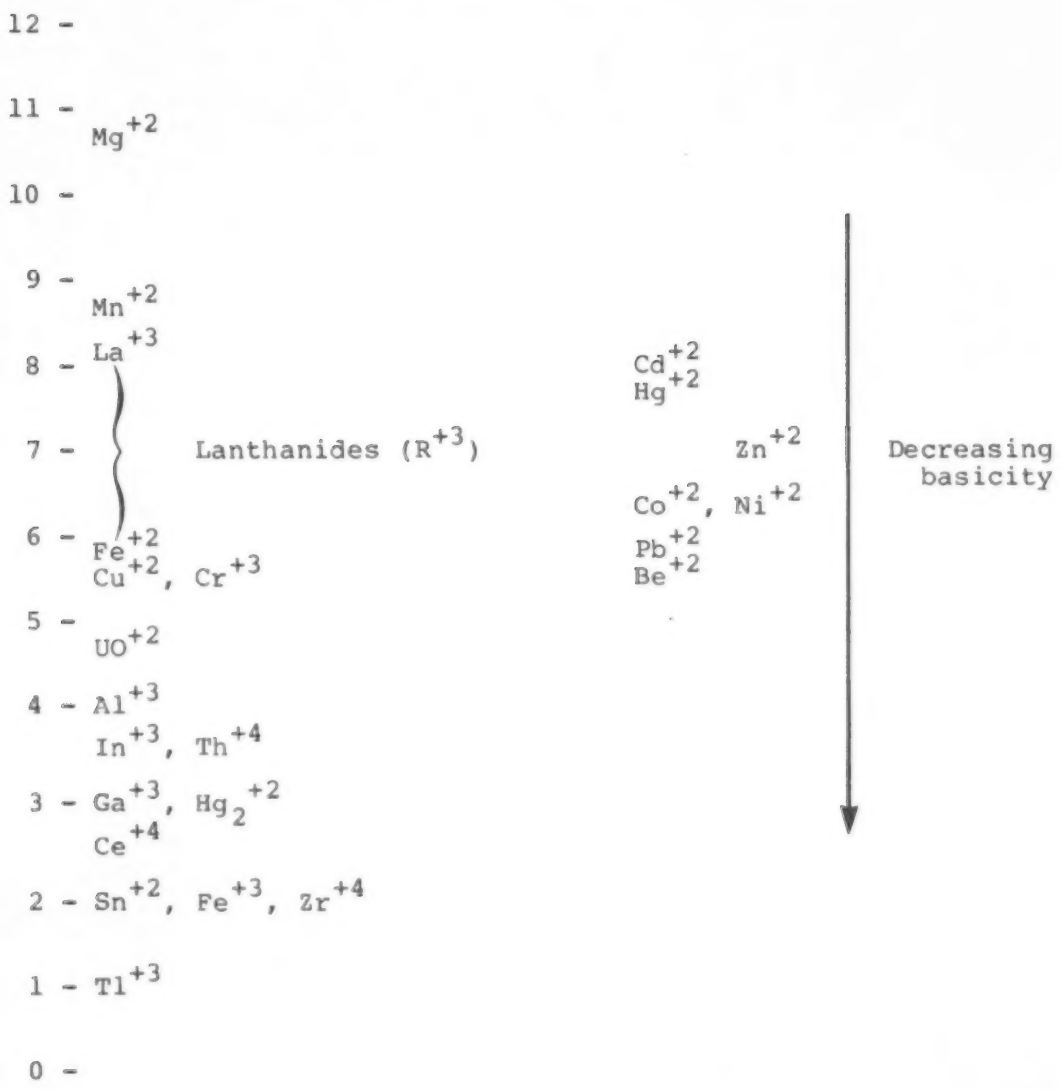
4. Rinse with (1-1) HNO_3. Rinse with tap water.

5. Rinse with (1-1) HCl. Rinse with tap water.

6. Rinse at least four times with D.I. water.

APPENDIX B

BASICITIES OF METAL IONS



Basicities of metal ions in terms of precipitation pH values for hydrous oxides and hydroxides.

Re: Inorganic Chemistry - 1957, T. Moeller - p. 502, John Wiley and Son, Inc.

APPENDIX C

PLANT WASTEWATER TREATMENT SYSTEMS

Plant

1 No information available. Process and wastewater treatment details were considered confidential by the plant management.

2 An integrated treatment system is used treating up to 2640 gph. Settling is present but not dewatering. No flocculent is used. NaOH is used as the precipitating agent.

3 Automatic flow-through system with NaOH used for precipitation. Sludge is dewatered by centrifuge to about 10 percent solids. System treats up to 7000 gph. Stripper dumps are bled into the treatment systems.

4 This plant has a finalizer automatic treatment system with some evaporative recovery on chrome. Lime is used for precipitation. Capacity of 35,000 gpd. Spillage containment is present.

5 A vacuum filter is used but no other information is available.

6 This plant utilizes lime from its vibratory finishing for precipitation. Cr^{+6} is treated in a packed Fe bed with H_2SO_4. A paper vacuum filter is used for dewatering. The system handles 28 gpm. HNO_3 and stripper are batch treated then metered to the treatment system.

Plant

7 NaOH is used to precipitate metals with a flow rate of 190 gpm. Dewatering after a Lamella™ type settling tank is accomplished with sand beds. Cr^{+6} and CN are treated separately then combined with acid/alkali wastewaters.

8 This plant treats 80,000 gpd using lime for precipitation. Underflow from a Lamella™ type settling tank is dewatered with a filter press. Some Cr is recovered by evaporation.

9 Wastewater at this plant is treated with aluminum sulfate and calcium chloride before being precipitated with NaOH. Dewatering is accomplished with an automatic plate and frame filter which dewaters to approximately 30 percent solids. The system has a capacity of 150,000 gpd.

10 This plant has five segregated treatment systems which sometimes run in series. The basic precipitation chemical is lime. The main system handles 3300 to 5400 gph. Dewatering is by gravity thickening.

11 An integrated system is used here with a dewatering bed. All spills are contained for batch treatment. NaOH is used for precipitation. Stripper dumps do not pass through the treatment system.

12 This plant uses lime for precipitation and a pressure filter for dewatering. Cr^{+6} and CN streams are segregated

Plant

and treated separately before combining with acid/alkali wastewaters. Capacity is 300 gpm.